JOURNAL

OF THE

AMERICAN CHEMICAL SOCIETY.

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VOLUME I.

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NEW YORK:
LEHMAIER & BRO., PRINTERS,
95 & 97 FULTON STREET.

1879.

540.5 A5 V.1

TABLE OF CONTENTS.

Volume I.

Nos. 1–3.

I.—Proceedings, January 2nd, 1879.

II.—A Method for the Detection of Artificial or Dextro-Glucose in Cane Sugar, and the Exact Determination of Cane Sugar by the Polariscope. By P. DE P. RICKETTS, Ph. D.. 2

III.—Proceedings, February 16th, 1879. 6

IV.—Relations Between the Temperature and Volume in the Generation of Ozone, with Description of a New Form of Ozonator. By ALBERT R. LEEDS, Ph. D. 8

V.—Upon the Titration of Hydrochloric Acid for Chlorine, and of Sulphuric and Nitric Acids for Hyponitric Acid. [2d Paper.] By ALBERT R. LEEDS. 18

VI.—On the Influence of Variations of Temperature on the Deviation of Polarized Light by Solutions of Inverted Sugar. By P. CASAMAJOR. 26

VII.—Proceedings, March 6th, 1879. 37

Additions to the Library of the American Chemical Society, from Jan. 1, to March 1, 1879. 39

List of Exchanges Received during January and February, 1879. 42

VIII.—Notes on Cupro-Manganese. By A. RAHT, E. M. 43

IX.—Examination of the Minnesota Early Amber Cane. By Prof. C. A. GOESSMANN. 44

X.—Notes Upon Chicle. By GEO. A. PROCHAZKA, Ph. D., and H. ENDEMANN, Ph. D. 50

XI.—Influence of Light upon the Decomposition of Iodides. By ALBERT R. LEEDS. 65

No. 4.

XII.—Proceedings, Thursday, April 3rd, 1879. 79

XIII.—Note on Arragonite. By H. A. WEBER. 79

Upon the Oxidation of Quinine by Means of Potassium Permanganate. By S. HOOGEWERFF and W. A. VAN DORP. Translated by GEO. A. PROCHAZKA, Ph. D.. 80

Report on the Progress of Analytical Chemistry, January to March, 1879. By GIDEON E. MOORE, Ph. D. 83

Abstracts from American and Foreign Journals.

American Chemical Journal, Vol. I., No. 1. Abstractor, J. P. BATTERSHALL. 111

Reports on American and Foreign Patents Relating to Chemistry.

Foreign Patents. Condensed from R. Biedermann's Report to the German Chemical Society, by H. ENDEMANN. 117

American Patents. Condensed from the Official Gazette of the U. S. Patent Office, by ARNO BEHR. 121

No. 5.

XIV.—Proceedings, May 1, 1879. 127

Additions to the Library of the American Chemical Society, from March 1 to June 1, 1879. 129

New Exchanges Received during March, April and May, 1879. 131

Report on the Berzelius Medal, Presented to the American Chemical Society by Dr. Hanbury Smith. 132

XV.—Analysis of Bindheimite from Arkansas. By CHAS. E. WAIT. 134

XVI.—Upon the Detection and Estimation of Nitrous Acids in Potable Waters, Acids, etc. I. With Meta-diamido-benzole; II. With Potassium Iodide. By ALBERT R. LEEDS 136

XVII.—Upon Ammonium Nitrite, and upon the By-products Obtained in the Ozonisation of Air by Moist Phosphorus. By ALBERT R. LEEDS. 145

Abstracts from American and Foreign Journals.

Berichte der Deutschen Chemischen Gesellschaft. Abstractor, P. TOWNSEND AUSTEN, Ph. D., F. C. S. 157

Paris Academy of Science. Abstractor, P. CASAMAJOR. 171

Reports on American and Foreign Patents Relating to Chemistry.

Foreign Patents. Condensed from R. Biedermann's Report to the German Chemical Society, by H. ENDEMANN. 174

American Patents. Condensed from the Official Gazette of the U. S. Patent Office, by ARNO BEHR. 177

Miscellaneous. 180

No. 6.

XVIII.—Proceedings, June 5th, 1879. 183
XIX.—Notes on the Chronology of the Isomeric Purpurines, and the Actual Relation of the Bodies which have been called by the names Anthrapurpurine, Isopurpurine and Flavopurpurine. By HENRY MORTON, Ph. D. 184
XX.—Petroleum and its Examination. By A. BOURGOUGNON. 188
XXI.—New Process for the Rapid Estimation of Pure Sugar in Raw and Refined Commercial Sugars. By P. CASAMAJOR. 205
XXII.—Contributions from the Laboratory of the Stevens Institute of Technology. By ALBERT R. LEEDS. 220
Abstracts from American and Foreign Journals.
Justus Liebig's Annalen der Chemie. Abstractor, GEO. A. PROCHAZKA, Ph. D. 233
American Patents. Condensed from the Official Gazette of the U. S. Patent Office, by ARNO BEHR. 260

No. 7.

XXIII.—Analysis of Air. By WM. VAN SLOOTEN, C. E. 263
Abstracts from American and Foreign Journals.
Bulletin de la Société Chimique de Paris. Abstractor, E. H. S. BAILEY, Ph. B. 271
American Chemical Journal. Abstractor, J. P. BATTERSHALL, Ph. D. 281
Berichte der Deutschen Chemischen Gesellschaft. Abstractor, P. TOWNSEND AUSTEN, Ph. D., F. C. S. 289
Journal fuer Praktische Chemie. Abstractor, EDGAR EVERHART, Ph. D. 293
Reports on American and Foreign Patents Relating to Chemistry.
Foreign Patents. Condensed from R. Biedermann's Report to the German Chemical Society, by H. ENDEMANN. 296
American Patents. Condensed from the Official Gazette of the U. S. Patent Office, by ARNO BEHR. 306
Miscellaneous 310

No. 8.

XXIV.—Additions to the Library of the American Chemical Society. 311
A Review of the Latest Investigations on the Dissociation of the Elements at High Temperatures. By H. ENDEMANN, Ph. D. 313
Report on the Progress of Analytical Chemistry, April to June, 1879. By GIDEON E. MOORE, Ph. D. 320
Miscellaneous. 381

No. 9.

XXV.—Proceedings, September 4th, 1879. 383
Abstracts from American and Foreign Journals.
Bulletin de la Société Chimique de Paris. Abstractor, E. H. S. BAILEY, Ph. B. 384
American Journal of Science and Arts. Abstractor, J. P. BATTERSHALL, Ph. D. 391
Berichte der Deutschen Chemischen Gesellschaft. Abstractor, P. TOWNSEND AUSTEN, Ph. D., F. C. S. 393
—Continued. Abstractor, J. H. STEBBINS, Jr., S. B. 395
Comptes Rendus de l'Academie des Sciences. Abstractor, A. BOURGOUGNON. 398
Reports on American and Foreign Patents Relating to Chemistry.
Foreign Patents. Condensed from R. Biedermann's Report to the German Chemical Society, by H. ENDEMANN. 404
American Patents. Condensed from the Official Gazette of the U. S. Patent Office, by ARNO BEHR. 405
XXVI.—Proceedings, October 2nd, 1879. 408

No. 10.

XXVII.—On a New Sulpho-Urea Compound. By JAMES H. STEBBINS, Jr., S. B. 415
XXVIII.—On Experiments with Fertilizers upon Sugar Cane, at Calumet Plantation, Bayou Téche, La. By Prof. C. A. GOESSMANN. 416
XXIX.—Contribution from the Chemical Laboratory of the Massachusetts Agricultural College. By Prof. C. A. GOESSMANN. 420
XXX.—Contribution to the Chemistry of Fruit Culture. By Prof. C. A. GOESSMANN. 423
XXXI.—Analysis of two Intestinal Calculous Concretions of Horses. By Prof. C. A. GOESSMANN. 430

No. 10. (Continued.)

XXXII.—Some Additional Notes on Ozone. By Albert R. Leeds, Ph. D..... 431
On the Density of Chlorine at High Temperatures. Note by Ad. Lieben (Comptes Rendus, No. 6, August 11, 1879). Translated by A. Bourgougnon..... 453
Report on American and Foreign Patents Relating to Chemistry.
Foreign Patents. Condensed from R. Biedermann's Report to the German Chemical Society, by H. Endemann..... 455
American Patents. Condensed from the Official Gazette of the U. S. Patent Office, by Arno Behr..... 457

No. 11.

XXXIII.—Proceedings, November 6th, 1879..... 461
XXXIV.—Some New Azo-Colors. James H. Stebbins, Jr., B. S..... 463
XXXV.—Action of Bone Black on Solutions of Pure Sugar. By P. Casamajor..... 468
XXXVI.—On the Formation of Compound Platinates, and a New Platino-Potassium Salt. By Lucius Pitkin..... 472
XXXVII.—Boracic Acid as a Preservative. By H. Endemann..... 477
On the Volatilization of Platinum in Chlorine Gas. By F. Seelheim. Ber. d. d. ch. Gesell..... 479
Answer to F. Seelheim's Remarks on My Experiments on Chlorine. By Victor Meyer. Translated by H. Endemann..... 481
Abstracts from American and Foreign Journals.
American Chemical Journal. Abstractor, J P. Battershall, Ph. D..... 482
Bulletin de la Société Chimique de Paris. Abstractor, E. H. S. Bailey, Ph. B. 486
Journal fuer Praktische Chemie. Abstractor, Edgar Everhart, Ph. D. 490
Reports on American and Foreign Patents Relating to Chemistry.
Foreign Patents. Condensed from R. Biedermann's Report to the German Chemical Society, by H. Endemann..... 498
American Patents. Condensed from the Official Gazette of the U. S. Patent Office, by Arno Behr..... 504

No. 12.

XXXVIII.—Proceedings, December 4th, 1879..... 507
XXXIX.—The Absorption of Sugar by Bone Black. By Henry A. Mott, Jr., Ph. D., E. M. 509
XL.—The Practical Determination of the Value of the Sugars of Commerce. By Henry A. Mott, Jr., Ph. D., E. M..... 514
XLI.—On the Preparation of Methylene Chloride, and Dioxyethyl-Methylene. By Wm. H. Greene, M. D..... 522
XLII.—Action of Benzo-Trichloride on Primary Amines. By James H. Stebbins, Jr., B. S. 524
XLIII.—Laboratory Notes. By H. Endemann, Ph. D., and Geo A. Prochazka, Ph. D. 525
Report on the Progress of Analytical Chemistry, July to September, 1879. By Gideon E. Moore, Ph. D..... 527
Reports on American and Foreign Patents Relating to Chemistry.
Foreign Patents. Condensed from R. Biedermann's Report to the German Chemical Society, by H. Endemann..... 587
American Patents. Condensed from the Official Gazette of the U. S. Patent Office, by Arno Behr..... 590

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JOURNAL

OF THE

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VOL. I. Nos. 1–3.

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ABSTRACTORS:

CONTENTS:

I. PROCEEDINGS, January 2nd, 1879.. 1
II. A Method for the Detection of Artificial or Dextro-Glucose in Cane Sugar, and the Exact Determination of Cane Sugar by the Polariscope. By P. DE P. RICKETTS, PH. D 2
III. PROCEEDINGS, February 16th, 1879. 6
IV. Relations between the Temperature and Volume in the Generation of Ozone, with description of a new form of Ozonator. By ALBERT R. LEEDS, PH. D..... 8
V. Upon the Titration of Hydrochloric Acid for Chlorine, and of Sulphuric and Nitric Acids for Hyponitric Acid. [2d Paper.] By ALBERT R. LEEDS. 18
VI. On the Influence of Variations of Temperature on the Deviation of Polarized Light by Solutions of Inverted Sugar. By P. CASAMAJOR. 26
VII. PROCEEDINGS, March 6th, 1879 ... 37
Additions to the Library of the American Chemical Society, from Jan. 1, to March 1, 1879..... 39
List of Exchanges received during January and February, 1879..... 42
VIII. Notes on Cupro-Manganese. By A. RAHT, E. M. 43
IX. Examination of the Minnesota Early Amber Cane. By PROF. C. A. GOESSMANN..... 44
X. Notes upon Chicle. By GEO. A. PROCHAZKA, PH. D., and H. ENDEMANN, PH. D 50
XI. Influence of Light upon the Decomposition of Iodides. By ALBERT R. LEEDS..... 65

NEW YORK:
LEHMAIER & BRO., PRINTERS,
162 WILLIAM STREET,
1879.

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THE AMERICAN CHEMICAL SOCIETY.

I.—Proceedings.

Regular Meeting, January 2nd, 1879.

The meeting was called to order at 8 o'clock, by Mr. Nichols, in the absence of the President and Vice-Presidents. Dr. Alsberg was elected Chairman. The minutes of the last meeting were read and adopted.

The resignation of Prof. Barker was read and accepted.

Messrs. H. M. McIntyre and G. W. Wigner were elected Members.

Messrs. F. W. Potter, Chas. de Lamothe, Chas. Marchand and Herbert Hazard were proposed as Members, and Messrs. C. F. Wingate and E. W. Leggett as Associates.

The Corresponding Secretary reported that the following circular has been sent to the proposed list of exchanges.

American Chemical Society,
No. 11 East 14th St.

New York, January 1, 1879.

The American Chemical Society, which was founded in April, 1876, has now completed the Second Volume of its Proceedings. The First Volume contains the papers read before this Society in 1876 and 1877, and the Second Volume those of 1878.

The papers read before this Society in 1879 will form Volume Three, which we propose to send to you as fast as the numbers appear, in exchange for the of the same year.

Please let me know if this proposal is agreeable to you.

In addition to our proceedings for 1879, we have the publications for 1876, 1877 and 1878, which we can exchange for the issued in those years, if you desire this exchange.

With much respect, I remain

Your obedient servant,

P. CASAMAJOR,

Corresponding Secretary.

Dr. Ricketts read the paper of the evening, entitled "A Method for the detection of artificial or dextro-glucose in cane sugar, and the exact determination of cane sugar by the polariscope."

In the discussion which ensued, Dr. Behr wished to know if Dr.

Ricketts had made any experiments in regard to the inverted sugar naturally present in raw and refined sugars. This inverted sugar, in most cases optically inactive at ordinary temperature, might have some action on light at 92° C, and would probably deviate the plane of polarization to the right. He also called attention to the fact that in certain raw sugars, as well as in different products of the refinery which contain naturally inverted sugar, the inverted sugar acts on polarized light in the same direction as cane and grape sugar. Such sugars, if tested after inversion in accordance with Dr. Ricketts' method, would not show zero, but more or less deviation to the right.

Mr. Ricketts said that in his experience the action by inverted sugar was zero at 92° C.

Mr. Casamajor called attention to the fact that beyond inverted sugar and aconitic acid none of the impurities of cane sugar are known. Beet sugars have been better studied. In them have been found asparagine, asparaginic and citric acids, dextrane, etc. Very likely, substances, the representatives of all these, as aconitic acid is the representative of citric acid, exist in cane sugars, and to them the irregularities are due.

Mr. Eastwick stated that he had also found a difference between the direct and inversion methods of testing in the lower products of the refinery, amounting sometimes to as much as two or three per cent.

The meeting then adjourned.

S. A. GOLDSCHMIDT,
Recording Secretary.

II.—A Method for the Detection of Artificial or Dextro-Glucose in Cane Sugar, and the Exact Determination of Cane Sugar by the Polariscope.

By P. DE P. RICKETTS, Ph. D.

Received February 25, 1879.

Having been called upon during the past Summer to examine a number of sugars for artificial or dextro-glucose, I found it necessary to determine some quick and accurate method for the detection of this substance, in the presence of invert and cane sugar. This, I find, can be readily accomplished, by taking advantage of the following facts:

1st. Artificial or dextro-glucose has a constant effect upon the plane of polarized light, at all temperatures under 100° C.

2d. Laevulose, or laevo-glucose, does not have a constant action upon the plane of polarized light; the amount of rotation to the left being diminished as the temperature is increased. See Watts' Dictionary of Chemistry, Vol V, page 464.

3d. Invert sugar, being a mixture of $\frac{1}{2}$ dextrose and $\frac{1}{2}$ laevulose, must, if the rotating power of the first to the right be constant, and that of the second to the left diminish as the temperature is increased, be neutral as to its effect upon polarized light, at somewhere near 90° C. See Watts' Dictionary of Chemistry, Vol v, page 465.

4th. Cane sugar, when acted on by dilute acids, is converted into invert sugar, while dextrose is unaltered.

At the suggestion of Prof. C. F. Chandler, who has favored me with his kind advice throughout, I enclosed the glass tube of the ordinary polariscope in a tin box, so arranged that it could be filled with water, and heated to any desired temperature under 100° C. I have upon the table, a Ventzke Soleil instrument, the central portion of which has been removed, and a tin box carefully adjusted in its place, which is fitted with a thermometer funnel and draining tube.

The ends of the box are fitted with brass caps, which contain circular pieces of clear plate glass; these caps screw on, and are perfectly water-tight. The glass tube containing the sugar solution is fitted with a second tube, having a small bore, which is ground in, and is sufficiently long to allow for any expansion of the liquid within, upon heating the apparatus.

To use the instrument, it is only necessary to fill the tube with the sugar solution to be tested, place it in the box, on rests, provided for the purpose, with the small glass tube in a vertical position, pour in distilled water until the box is $\frac{3}{4}$ full, and then apply heat from below, raising the temperature very gradually. I examined first a series of raw and refined sugars of various grades, and found that after clarification, inversion, &c., they read, almost without exception, zero, at ninety-two degrees Cent.

The effect of the temperature upon the readings can best be shown by example, and I therefore give a few of the results obtained in testing pure cane sugar.

SUGAR.		TEMPERATURE, CENTIGRADE.				
No.	Grade.	60°	70°	80°	90°	92°
1.	CUBA HHDS	—11	—7	—4.1	—1	0
2.	MUSCAVADO	—10.1	—7.4	—5	—0.5	+0.2
3.	MEXICAN	—11	—6.8	—3.5	—0.8	0
4.	REFINED CUT-LOAF.....	—10	—7.5	—3.6	—0.7	0
5.	" X C.....	—10	—7.5	—4	—0.5	0
6.	" X SUGAR.....	—9.5	—7	—4	—0.5	+0.3
7.	" X LOW GRADE.	—13.7	—9.3	—5.5	—1.0	0

I next examined a number of samples of commercial glucose, and found that after preparing them as in case of the sugars, that they did not vary in their effect upon the plane of polarized light, the same sample of glucose giving a constant rotation to the right, at all temperatures under 100° C.

I give below a few of the readings obtained from some of the samples tested, which did not, however, contain in each case the same amount of chemically pure glucose, as that determined by Fehling's test.

SAMPLE.		TEMPERATURE, CENTIGRADE.					
	Marks, etc.	60°	70°	80°	90°	92°	
No. 1.	CORN GLUCOSE, Am. Grape Sugar Co.	+87	+87	+87	+87	+87	&c.
" 2.	GLUCOSE, Locality Unknown.	+35	+35	+35	+34.5	+34.3	&c.
" 3.	DRY GRAPE SUGAR.	+56	+56	+56	+56	+56	&c.
" 4.	GRAPE SUGAR, Tromsdorff.	+35	+35	+35	+35	+35	&c.

Nos. 2 and 4 contained the same amount of chemically pure glucose.

By observing a number of samples of commercial glucose, I found the average rotation to the right at 92° C. to be 87 divisions of the scale of the instrument, when the sample tested contained 63 per cent. by Frehlings test, of chemically pure glucose. Hence, if 26.048 gms. be the amount taken for observation, $26.048 \times \frac{63}{100} \div 87 \times 100 = 18.864$ gms., is the amount of dry, chemically pure, glucose necessary to read 100 divisions on the scale, or each division is equal to 0.1886 gms. of chemically pure glucose.

Having established my data as to the action of invert sugar and dextro-glucose upon polarized light, and the value of the divisions on the scale of the instruments, I made a series of mixtures of commer-

cial glucose, with cane sugar of various grades, and found I could determine with accuracy the amount of glucose added, even where the mixture contained as little as one and one-half per cent. of commercial glucose. The following readings, made with two mixtures, prepared by my assistant, and submitted to me without my knowing the percentage of adulteration, will, I think, be sufficient to illustrate the method and prove its value.

SAMPLE.		TEMPERATURE, CENTIGRADE.				
	Contained Chemically Pure Glucose.	60°	70°	80°	90°	92°
No. 1.	6.6 grms.	+26.7	+28	+32	+34	+35
" 2.	8.2 "	+38	+41	+41.9	+42.8	+43

	ROTATION OBSERVED.		VALUE OF SCALE.		GRMS. FOUND.
No. 1.	+ 35	×	0.1886	=	6.3 grms.
" 2.	+ 43	×	0.1886		8.185 "

Of course, in testing the mixtures, care was taken to invert all the cane sugar before observation in the instrument.

By weighing out 18.86 gms. of the invert sugar present, the second will give the cane sugar and dextro-glucose; the third, the dextro-glucose.

The temperature given, 92° C., refers to the water bath; the temperature of the solution within the tube, may be slightly lower, but as the same apparatus has been employed throughout, the results are comparative, and will hold true.

To adjust the instrument for my experiments, I test it with, first, a solution of pure cane sugar; second, a solution of pure cane sugar inverted; third, a solution of standard commercial glucose, and fourth, a mixture containing a known percentage of pure glucose.

The first solution should read 100; the second zero at 92° C.; the third, indicate a constant dextro-rotation, and the fonrth, read to the right. After inversion, according to the amount of dextro-glucose added, when observed at 92° C., or the temperature at which the solution of invert sugar read zero, the amount of dextro-glucose contained in the sample to be tested will read percentage on the scale, and the calculation given above be avoided.

In order to determine the effect of raising the temperature on the readings of a solution of cane sugar, I took 26.048 grms., clarified, and observed it in the instrument. I found that the reading at 92° C. would be slightly less than at the ordinary temperature, especially if the sugar was in the slightest degree acid; but if a slight excess of carbonate of soda or lime be added to the sugar solution before fill-

ing the polariscope tube, the readings would be nearly constant. Hence, to determine invert cane sugar and dextro-glucose in a sample, observe first, at—say 25° C.; second, at 92° C.; without inverting; third, invert and read at 92° C. The first observation would give us the cane sugar and the dextrose less the left rotation due to laevulose.

I am still experimenting on sugars of various grades, and may be able to give you in another paper some further details.

In conclusion I wish to express my thanks to my assistant, Mr. C. L. Constant, who has aided me materially in the experimental work necessary for the preparation of this paper.

III.—Proceedings.

Regular Meeting, February 16, 1879.

The meeting was called to order at 8:15 o'clock, Vice-President Leeds in the chair. The minutes of the last meeting were read and adopted.

The Board of Directors reported as follows:

At a meeting held January 16, 1879—*Present*, Messrs. Leeds, Nichols, Casamajor, Miller, Waller, Endemann and Goldschmidt.

The Treasurer reported a balance of $984.79.

The following bills were audited and ordered to be paid: T. J. Fay, $9.00; G. E. Stechert, $19.65.

On motion of the Treasurer it was resolved to appoint a committee to mature a plan of action for the collection of the amount due the Society for arrears of dues, and to report the names of members to be dropped from the rolls of the Society for such non-payment. The chair appointed as committee Messrs. Casamajor, Nichols and Goldschmidt.

Resolved, That no one should be considered a member of the Society, or receive the proceedings or their publications, until he shall have paid his initiation fee and dues for the current year.

Resolved, That the Librarian hereafter send out the proceedings.

Resolved, That the Treasurer be empowered to make such arrangements in regard to re-engaging the rooms of the Society as he may deem proper.

At a meeting of the Board of Directors, held February 6, 1879—*Present*, Messrs. Leeds, Goldmark, Endemann, Miller, Nichols, Waller, Casamajor, Eastwick and Goldschmidt.

The following bills were audited and ordered to be paid: G. A. Kundahl, $52.00; G. E. Stechert, $6.60; Baker & Godwin, $87.87; S. A. Goldschmidt, $17.25.

The Treasurer reported a balance of $1,100.00.

The Committee on Publications were authorized to publish the proceedings for the coming quarter. They were also directed to publish Vol. I, No. 4, by March 1, 1879.

The Committee on delinquents reported 104 members in arrears, of whom 42 had never paid one cent, and that they had sent the following circular to these gentlemen:

AMERICAN CHEMICAL SOCIETY,
No. 11 East 14th St.

NEW YORK, January 24, 1879.

SIR: The Committee on Delinquents, of the American Chemical Society, has directed me to inform you that you owe to this Society the amount specified in the enclosed notice. By decision of this Committee, the names of those who have not paid their dues for the years prior to 1879, shall be dropped from the roll of membership on the first day of May next. We hope that you will give this matter your immediate attention, as we would regret the necessity of dropping your name from our rolls.

With much respect, I remain
Your obedient servant,
P. CASAMAJOR,
Corresponding Secretary.

Messrs. F. W. Potter, Chas. de Lamothe, Chas. Marchand and Herbert Hazard were elected Members, and Messrs. E. W. Leggett and C. F. Wingate Associates.

Messrs. C. G. Wheeler, T. B. Stillman, August Eimer, —— Erhardt, W. F. Hildebrand and John F. Kelly were proposed as members, and Messrs. A. W. Hazard, Joseph Binnus, John Seal and Wm. Ziegler as Associates.

The resignations of Messrs. H. M. Drowne, A. Hallgarten, E. L. Kalbfleisch, J. H. Rickettson, J. L. Morgan, Jr. and Davenport Fisher were read and accepted.

Dr. Endemann taking the chair:

Prof. Leeds read the first two papers, viz: 1. "An algebraic expression for the curve expressing the relation between temperature and volume in the generation of ozone." 2. "Quantitative

measurements of the chemical energy of the solar ray." The papers were accompanied with photographic and experimental illustrations.

In discussing the second paper, Dr. Endemann remarked that Grotthuss had studied the action of the various rays upon starch iodides, and had found that the red and yellow rays bleached this substance, while the violet ray restored the color. The possibility of a greater energy of the violet ray at noon would correspond with the results obtained by Prof. Leeds.

The Vice-President resuming the chair.

Mr. Sherer read a paper entitled "A Method for the Determination of Dextro-glucose and Cane Sugar in Refined Sugars and Syrups,"* which was discussed by Dr. Behr.

Mr. Casamajor then read a paper "On the Influence of Variations of Temperature, and the Deviations of Polarised Light by Solutions of Inverted Sugar." After which the meeting adjourned.

S. A. GOLDSCHMIDT,
Recording Secretary.

IV.—Relations between the Temperature and Volume in the Generation of Ozone, with description of a new form of Ozonator.

By ALBERT R. LEEDS, Ph. D.

The methods usually employed to generate ozone by means of the slow oxidation of phosphorus, partly immersed in water, are quite unsatisfactory. Thus Miller (Elements of Chemistry, 2d. Ed., Part II., p. 23) directs that a stick of clean phosphorus, moistened with a few drops of water, should be placed in a bottle of atmospheric air. In an hour or two the production of ozone attains a maximum, when, if the phosphorus be not removed, the ozone disappears, owing to its combination with the phosphorus. Instead of a bottle, a large glass balloon is preferably employed, which Arendt (Lehrb. der Anorgan. Chem., p. 416) directs to be covered with a glass plate, and allowed to remain before using for twelve hours. Gorup-Besanez (Anorgan. Chem., p. 358) recommends, in addition to the foregoing, that the balloon should be maintained for several hours at a temperature of 16° to 20°. As means of studying, or even of exhibiting to a class, the properties of ozone, everyone who has used these and similar devices, will probably have found them disappointing. At times a

* This paper has been withdrawn by the author, C. P. P.

considerable evolution of ozone occurs; at others, little, or none. The causes of these variations will appear on examination of the experiments detailed later.

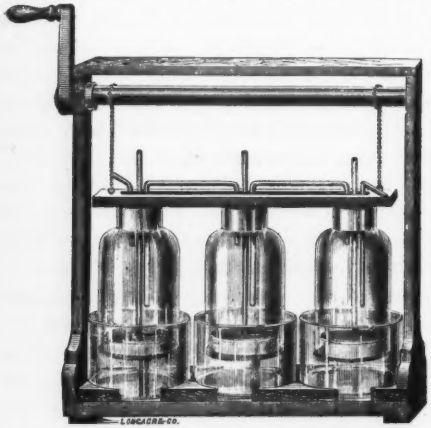
In a work published much earlier than the two last quoted, an apparatus is described and illustrated (Lecons de Chimie, A. Riche, Tome I., p. 106), which was a step in the right direction. The balloon was provided with an entrance and exit tube, the latter extending nearly to the bottom, so that a slow continuous current of air might be drawn through by means of an aspirator. The ozonized air was washed in the usual manner, and the liquids to be subjected to the action of ozone were placed in a second wash-bottle. This contrivance was adopted, a large bell-jar turned upside down and covered with a glass plate, being substituted for the wash-bottle, when it was desired to ozonize dyed goods, flowers and similar large objects. The apparatus was further improved by using a bell-jar in place of the balloon. The bell was set in a dish containing a number of sticks of phosphorus, the water with which they were partly covered acting as a seal. The disagreeable operation of occasionally cleansing the phosphorus, by scraping, was obviated by immersing them before using in a mixture of potassium bichromate solution and sulphuric acid.

These improvements, however, were of small importance compared with that effected by substituting the bichromate mixture for the water in the generator itself. The scanty, fitful evolution of ozone was replaced by a copious and constant supply. The dish was now replaced by a jar capable of holding a considerable amount of the bichromate solution, and by permitting a considerable change of level in the liquid, the air could either be drawn through by aspiration or forced through by pressure. Instead of placing the phosphorus on the bottom, the sticks were supported on a glass disc, which dropped into a cell connected with a paraffined iron rod sliding up and down, through the cork at top. In another generator, the cell was replaced by a brass stirrup, electroplated with gold; but this had to be abandoned, the solution speedily eating away the metal.

A difficulty now arose from the danger of inflammation, resulting from the great energy of oxidation. This danger seemed largely due to irregular melting down of the phosphorus, ridges being formed which protruded too far above the liquid. This unequal action also greatly diminished the amount of surface which could be safely exposed. To obviate these difficulties, watch-glasses were placed in a shallow tin dish filled with water, sufficient fragments of phosphorus placed in dish, and the temperature raised until, by melting of the phosphorus, a num-

ber of rounded cakes of uniform size had been made. Six of these could be placed upon the stage at one time, and the stage adjusted so that each cake exposed about 9 c.m.^{2} of surface area. As the convexity of the exposed portions was small, the liquid, by surface action, was constantly drawn over it in thin streams, and not only kept the phosphorus wet, but always clean and in condition of maximum chemical activity.

It was found, as will be seen by results stated below, that the air ozonized in one bell, could have its ozone percentage notably increased by passing through a second bell similar to the first, through a third, and so on. But as the manipulation of three, or even two bells, connected with glass tubes, was troublesome, it became necessary to arrange suitable machinery. The bells were, therefore, cemented into heavy brass caps. Through these caps and the paraffined cork beneath, connecting tubes of heavy glass were passed, and a sliding glass rod, which terminated below in a horizontal circle. The latter carried a disc of sheet lead, with a slot permitting its removal. The caps were then screwed fast to a board having a suitable opening down its middle to permit of passage of tubes, etc., and this again fastened to chains by which it might be readily raised or lowered. The frame is made of such height that the jars may be easily slipped from under the bells when desirable.* Thermometers are fastened into the side of each bell, their bulbs dipping just below the surface of the liquid.



It was evidently essential to convenient working of this apparatus to have a flexible connection between exit tube and wash bottle. But herein arose a very serious difficulty, India-rubber tubing being destroyed. Through suggestion of Prof. Silliman, and by kindness of Mr. A. G. Day, the patentee, I became provided with a great variety

* These ozonators are manufactured by S. Hawkridge, successor to Wales & Co., Stevens' Institute of Technology, at the price of thirty dollars.

of kerite tubing. Weighed samples of each kind were subjected to equal amounts of ozone for equal intervals, their changes and the products of decomposition noted. Without detailing these experiments, suffice it to say that the specimens which gained most in weight underwent greatest decomposition, some indeed crumbling to pieces, while others which gained none, also did not change in appearance or physical characters. Of these, the most satisfactory was selected, and from it as a guide Mr. Day manufactured the ozone-resisting kerite, which is now used in connection with the ozonator, and generally during the progress of this investigation.

The improvements effected in the ozone-generating apparatus, were due, as has been said above, to a series of quantitative trials, the results of each set of experiments suggesting modifications in the next series. In the first place, phosphorus was used with water alone, one bell only being employed. The ozonized air was washed, and afterwards drawn through two Peligot tubes, containing a ten per cent. solution of potassium iodide. The second tube was added as a guard, but in practice is unnecessary, complete absorption occurring in the first. The liberated iodine, after acidifying with sulphuric (free from nitrous) acid, was titrated with sodium hyposulphite. In each experiment, 9 liters of air were drawn over. It soon became evident that widely differing results were obtained at different temperatures, the percentage of ozone falling off with decrease of temperature, to nought. Starting from this point, it appeared to increase, according to some unknown law, to a maximum, and then decreased again with further increase of temperature, until the point of inflammation of the phosphorus was attained. The volume ratio is given in terms of 0.005 Vpc. taken as a convenient unit :—

I.—OZONE FROM PHOSPHORUS IN WATER.

T	Mgrm. per Liter air.	Wpc.	cc per Liter.	Volume Ratio.	Wpc. of O.	Vpc. of O.
2°	none		none			
19°.5	0.615	0.0478	0.290	5.8	0.2065	0.139
25°	0.882	0.0682	0.413	8.3	0.2946	0.198
25°	1.050	0.0812	0.494	9.9	0.3508	0.237
30°.5	0.326	0.0252	0.153	3.1	0.1088	0.073

For convenience of comparison between these experiments and others, in which oxygen, not air, has been ozonized, columns VI and

VII have been added, the former giving the percentage by weight of ozone in the oxygen passed over, disregarding the nitrogen, the latter the percentage by volume. It will be seen that the maximum was obtained at about 25°, when the air contained 1 mgrm. ozone in the liter, or about one-half cubic centimeter.

In the second series of experiments, the water was replaced by a solution, containing to the liter of saturated solution potassium bichromate, 150 cc. H_2SO_4. In each experiment, $8\frac{1}{2}$ liters air were drawn over. The results were as follows:

Temperature.	Time.	Hyposulphite.
7°		0.40 c.c.
13°—14°	70 minutes	4.25
17°—20°	56 "	6.77
21°—22°		7.10
24°	60 "	8.35
30°—31°.5	60 "	7.13
31°—32	60 "	7.18

From which it would appear that at a temperature of about 6° no ozone is given off, and from this point the percentage rises, until the temperature attains about 24°, the percentage falling off quite evenly on both sides of the maximum. The maximum production was 1.86 mgrm. per liter air, corresponding to 0.87 cubic centimeter.

II.—OZONE FROM PHOSPHORUS IN BICHROMATE (ONE BELL-JAR).

T	Mgrm. per Liter air.	Wpc.	cc per Liter.	Volume Ratio.	Wpc. of O.	Vpc. of O.
7°	0.089	0.0069	0.042	0.8	0.0298	0.020
13°.5	0.945	0.0730	0.441	8.8	0.3154	0.210
20°	1.505	0.1163	0.705	14.1	0.5011	0.336
21°.5	1.580	0.1221	0.741	14.8	0.5270	0.353
24°	1.857	0.1438	0.870	17.4	0.6221	0.415
31°.5	1.590	0.1229	0.745	14.9	0.5314	0.355

A third series of experiments was instituted to determine whether the air which had been ozonized in this manner, could receive an increment of ozone by being again subjected to the influence of phosphorus in a second bell-jar. The liquid contained to each liter of saturated solution of bichromate, 250 c.c. H_2SO_4.

Temperature.	Amount of Air.	Time.	Hyposulphite.
13°	2 liters.		1.20 c.c.
19°	2 "	12 minutes.	2.00 "
21°	2 "		2.1 "
23°	3 "		3.0 "
24°	3.5 "	20 minutes.	4.0 "
24°	2 "		2.6 "
27°	2 "		2.25 "

III.—OZONE FROM PHOSPHORUS IN BICHROMATE (TWO BELLS).

T	Mgrm. per Liter air.	Wpc.	cc per Liter.	Volume Ratio.	Wpc. of O.	Vpc. of O.
13°	1.134	0.0877	0.531	10.6	0.3789	0.255
19°	1.890	0.1460	0.885	17.7	0.6307	0.425
21°	1.985	0.1546	0.929	18.6	0.6696	0.446
23°	1.890	0.1460	0.885	17.7	0.6307	0.425
24°	2.155	0.1701	1.009	20.2	0.7344	0.489
24°	2.457	0.1933	1.151	23.0	0.8338	0.552
27°	2.127	0.1643	0.996	19.8	0.7098	0.478

It will be seen that the air ozonized in two bells, likewise contains a maximum percentage at 24°, but this maximum is 25 per cent. higher than the corresponding amount obtained with one bell; and, in general, the percentage obtained by two bells is 25 per cent. greater than that with one bell at any given temperature.

Before proceeding further, it appeared advisable to determine whether some other liquid might be substituted for the bichromate with advantage. Alkaline liquids seemed inapplicable, owing to the danger of formation of alkaline hypophosphites, and the evolution of hydrogen phosphide. Of the acids, sulphuric appeared the most suitable, although even this was attended with the disadvantage of the probable formation of some sulphurous anhydride at the same time. Mixtures of potassium permanganate, and similar salts with dilute acid, have not as yet been tried. The permanganate would have the disadvantage of being expensive, and even when sold as chemically pure, is usually contaminated with much potassium chlorate. The results obtained with a bath containing 250 c.c. H_2SO_4 to the liter of water, were as follows, two liters of air aspirated in each experiment, the interval varying from 12 to 25 minutes:

Temperature,	25°	Sodium Hyposulphite,	1.60	c.c.
"	26°	"	2.90	"
"	25°	"	1.06	"
"	26°	"	1.30	"
"	26°	"	1.35	"

These low results made it appear probable that the phosphorus had become oxidized. It was, therefore, allowed to stand for some time in the bichromate mixture, and the experiment repeated:—

Temperature, 27° Sodium Hyposulphite, 1.70 c.c.

Although even this amount was not so high as that previously obtained under similar circumstances, at the same temperature, when the bichromate was employed, yet it confirmed the justness of the above supposition, and caused the use of the acid alone, as a bath, to be definitely abandoned. In the following table, the trials are given in the order as made, with the view of showing the gradual deterioration. The abnormally high result obtained for the first 26°, was due to the phosphorus having been kept for an unusual length of time in contact with the air in the bells, before aspiration was begun.

IV.—OZONE FROM PHOSPHORUS IN SULPHURIC ACID (TWO BELLS).

T	Mgrm. per Liter air.	Wpc.	cc per Liter.	Volume Ratio.	Wpc. of O.	Vpc. of O.
25°	1.512	0.1169	0.708	14.2	0.5011	0.340
26°	2.741	0.2119	1.283	25.7	0.9154	0.616
25°	1.002	0.0775	0.469	9.4	0.3347	0.225
26°	1.229	0.095	0.575	10.5	0.4104	0.276
26°	1.276	0.0986	0.597	11.9	0.4259	0.287
27°	1.607	0.1242	0.752	15.0	0.5365	0.361

Another related query was, whether so concentrated a bath was essential. To settle this point, a solution was made containing to each 1250 c.c. H_2O, 150 c.c. H_2SO_4, and 25 grms. $K_2Cr_2O_7$. The determinations were purposely made at about the maximum temperature:

Temperature,	26°5	Sodium Hyposulphite,	2.00	c.c.
"	26°5	"	2.18	"

V.—OZONE FROM PHOSPHORUS IN DILUTE BICHROMATE (TWO BELLS).

T	Mgrm. per Liter air.	Wpc.	cc per Liter	Volume Ratio.	Wpc. of O.	Vpc. of O.
26°.5	1.890	0.1460	0.885	17.7	0.6307	0.425
26°.5	2.060	0.1592	0.965	19.3	0.6868	0.461

These results, though inferior to those obtained with the more concentrated solution, were nevertheless so satisfactory and constant, that this mixture was adopted, and used during the subsequent work.

The results arrived at on increasing the number of bells to two had been so gratifying, that it was thought advisible to employ three, and to combine them in an arrangement which would permit them to be easily handled, the form finally adopted being that figured in the text. The temperatures given are those read from the thermometers in the three bells, a reading being taken from each at the beginning, middle and end of every experiment, and the average result stated. After the apparatus and bath had arrived at temperature desired, the stages were raised by one motion of crank, to height proper to expose a uniform surface of phosphorus, eight liters of air aspirated, and then one, two or more liters, drawn over, and the ozonized air titrated by solution of potassium iodide. All the figures obtained are given, although they are not so uniformly progressive as was hoped for, a result due perhaps in part to the difficulty of completely changing the atmosphere in three six-liter bells by a slow current of air, without an expenditure of an amount of time which other duties would not permit.

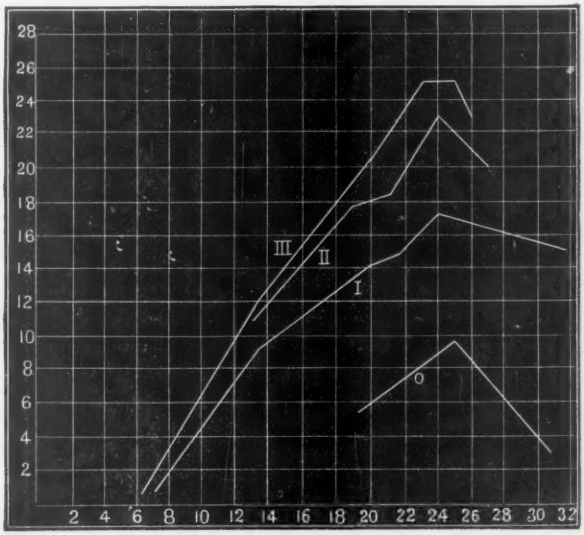
Temperature.	Amount of Air.	Time.	Hyposulphite per Liter.
6°.3	3 liters.	15 minutes.	0.033 cc.
6°.3	4 "	20 "	0.033 "
10°.63	2 "	15 "	0.08 "
11°.1	2 "	13 "	0.10 "
11°.21	2 "	15 "	0.20 "
12°.22	2 "	15 "	0.225 "
12°.22	2 "	13 "	0.215 "
13°.33	2 "	20 "	0.68 "
13°.33	2 "	10 "	0.60 "
14°.99	2 "	15 "	0.40 "
14°.99	2 "	15 "	0.40 "

Temperature.	Amount of Air.	Time.	Hyposulphite per Liter.
15°.88	2 liters.	15 minutes.	0.42 cc.
16°.11	1 "	10 "	0.66 "
16°.11	1 "	10 "	0.68 "
17°	2 "	15 "	0.84 "
17°.5	3 "	18 "	0.61 "
20°	2 "	18 "	1.15 "
22°.78	2 "	15 "	1.52 "
22°.78	2 "	10 "	1.32 "
25°	2 "	15 "	1.42 "
26°.11	2 "	13 "	1.30 "
26°.11	2 "	10 "	1.20 "

VI.—OZONE FROM PHOSPHORUS IN DILUTE BICHROMATE (THREE BELLS).

T	Mgrm. per Liter air.	Wpc.	cc per Liter.	Volume Ratio	Wpc. of O.	Vpc. of O.
6°.3	0.0624	0.0048	0.029	0.6	0.0208	0.014
10°.63	0.1512	0.0116	0.071	1.4	0.0501	0.034
11°.1	0.1890	0.0146	0.089	1.8	0.0631	0.043
11°.21	0.378	0.0292	0.177	3.5	0.1262	0.085
12°.22	0.416	0.0321	0.195	3.9	0.1391	0.093
13°.33	1.285	0.0993	0.602	12.0	0.4290	0.289
13°.33	1.134	0.0876	0.531	10.6	0.3784	0.255
14°.99	0.756	0.0587	0.354	7.1	0.2536	0.170
15°.88	0.794	0.0618	0.372	7.4	0.2678	0.179
16°.11	1.285	0.0993	0.602	12.0	0.4290	0.289
17°	1.588	0.1229	0.745	14.9	0.5309	0.355
17°.5	1.153	0.0889	0.540	10.8	0.3840	0.259
20°	2.170	0.1677	1.018	20.4	0.7245	0.486
22°.78	2.684	0.2075	1.257	25.1	0.8964	0.603
25°	2.684	0.2075	1.257	25.1	0.8964	0.603
26°.11	2.457	0.1902	1.151	23.0	0.8212	0.552
26°.11	2.268	0.1778	1.062	21.2	0.7690	0.510

It will be seen from the above, and from the accompanying diagram which graphically represents the relations between the tem-



peratures and the corresponding volumes, that in each case the maximum volume was obtained at about 24° C. While there is a general similarity in the curves, yet it will be noted that III does not fall relatively so far beyond II, as II beyond I, and there would accordingly be little advantage in increasing the number of bells beyond three. Below 6° C. no ozone appears to be generated, and probably little or none beyond 38°, phosphoric oxide being formed in increasing quantities as the temperature approaches 44°, the melting-point of phosphorus, and the danger of inflammation correspondingly increasing. On substituting certain of the values determined for curve I, in the general formula, it was found that they satisfied the equation for the oblique hyperbola of the primary order. This curious coincidence is noted, although we are not able to attach to it a meaning of scientific import. But the practical value of these curves has been constantly tested in the course of protracted experiments, and it has been possible to determine in each case to what extent the substance under examination has been subjected to the action of ozone. And while there is little over 2½ mgrms. of ozone in the liter of air drawn

over, at the maximum, yet since, by attaching the ozonator to the water-pump, it may be allowed to act during days and nights for several days without further attention, the yield of ozone may be indefinitely increased.

Since writing the above, attempts have been made to improve the ozonator in various details. It was thought that it might be simplified by substituting for the jars a rectangular trough of copper, lead, or wood painted inside with asphalt. The copper was rapidly attracted by the chromic acid (56 p. c. of copper in the form of turnings went into solution over night), and the asphalt likewise. In the case of lead, it was found that 16.543 grm. turnings increased in weight to 16.546 grms., on standing in the mixture 20 hrs., so that the superficial crust of insoluble salts formed would serve for protection. But its weight, if made thick enough to keep the shape, was objectionable. Moreover, it was important to make the trough of a material which could be heated directly by gas-jets, when it was desired to bring the apparatus to the temperature of maximum activity, or 24° c. This has been done by the addition of a suitable copper water-bath, in which the jars are placed, provided with proper arrangements for heating. Manifestly, the same bath will serve for cooling, when the summer temperature shall exceed 24°.

It gives me great pleasure to acknowledge the co-operation of my assistant, Dr. Edgar Everhart, in the performance of the foregoing experiments.

V.—Upon the Titration of Hydrochloric Acid for Chlorine, and of Sulphuric and Nitric Acids for Hyponitric Acid. [2d Paper.]

By ALBERT R. LEEDS.

In a former paper I have given the results obtained on titrating eight specimens of so-called pure acids, the titrations beginning October 27th and continued, as notable amounts of starch iodide were formed, until December 5th. At this time solutions VI, VII and VIII were thrown away; the others were titrated giving the following amounts of standard sodium hyposulphite:

	I.	II.	III.	IV.	V.
Dec. 12th....	None	0.34 cc	1.04 cc	0.10 cc	0.10 cc
Jan. 3rd ...	None	0.75	3.25	2.00	0.50
Totals.....	1.23 cc	4.91 cc	9.46 cc	5.70 cc	3.01 cc

It will be seen from column II, that the 5 cc of HCl in 1 liter water, had liberated nearly five times the amount of iodine as the 1 cc HCl under like circumstances (column I). The sulphuric acid (columns III and IV) had effected far more decomposition than the nitric (V), and this again much more than the hydrochloric acid of similar strength (I). The significance of these differences were not known when the five solutions were thrown away (Jan. 3rd), but the cause of the combined change was, having become known through the following experiment.

In place of solutions VI, VII and VIII, six two-liter bottles were taken, and 1000 cc water, 4 cc starch solution, and 1 cc KI solution of 10 per cent. added to each. Three of these, after addition of 1 cc H_2SO_4, 1 cc HNO_3 and 1 cc HCl respectively (same acids as in columns VI, VII, VIII), were placed in diffused light; the other three, similarly treated, were set in the dark (Dec. 6th).

TITRATION OF ACIDS.

	Dark.			Light.		
	H_2SO_4	HNO_3	HCl	H_2SO_4	HNO_3	HCl
Dec. 9th....	0.13 cc	0.30	0.13	0.96	0.79	0.49
Jan. 3rd....	None	None	None	4.70	3.00	3.20
Jan. 4th.....						

Those in the dark having ceased to change, three similar new solutions were substituted:

Jan. 6th....	0.033	0.03	0.03			
Jan. 20th...	None	None	None	3.60	2.50	1.50

The three new solutions having ceased to change were thrown away; those in the light were exposed to *sun-light* for six hours.

Jan. 20th, (6 hrs.).....	0.50	0.40	0.20

The last experiment was made in consequence of the results obtained in several preceding trials. Eight bottles, each containing 1 cc H_2SO_4, 3 cc starch solution and 1 cc KI, were partly filled with various amounts of water, and four placed in light, four in darkness. After 24 hrs. they were titrated (column I). This experiment was repeated (column II); in this case, the same solutions being again titrated after the lapse of 5 days:

TITRATION OF 1 cc H_2SO_4—(1.845 Sp. Gr.)

Sun-light.

	I.	II.	III.
With 100 cc H_2O	0.18 cc	0.23 cc	1.20 cc
" 50 cc "	0.15	0.17	0.98
" 25 cc "	0.23	0.23	1.06
" 20 cc "	0.20	0.20	1.00

Darkness.

	I.	II.	III.
With 100 cc H_2O	Trace.	0.03 cc	0.10 cc
" 50 cc "	0.06 cc	0.06	0.20
" 25 cc "	0.15	0.10	0.30
" 20 cc "	0.23	0.18	0.30

An inspection of these figures developed the curious fact, that in the dark, the amount of decomposition of the potassium iodide, was approximately proportional to the degree of concentration. In the light, similar solutions appeared to obey no similar law, or indeed any law of a definite character. To determine to what degree of dilution the above law held true, a number of bottles, each containing 2 cc KI solution, and 5 cc starch, but with various amounts of water, were placed, some in sun-light, others in darkness, allowed to stand over night, and titrated at the end of 24 hours.

TITRATION OF 1 cc H_2SO_4—(1.845 Sp. Gr.)

	Sun-light.	Darkness.
With 500 cc H_2O.		Indeterminable.
" 400 " "	0.70 cc	"
" 300 " "		"
" 250 " "		"
" 200 " "	0.56	0.01 cc
" 150 " "		0.006
" 100 " "	0.44	0.06
" 75 " "		0.15
" 50 " "	0.30	0.06
" 25 " "		0.80
" 20 " "	0.30	

This experiment brought out the fact that in sun-light, the action was the reverse of that in darkness, although the law governing the rate of change did not directly appear. In the dark, it appeared that at ordinary temperatures, no appreciable amount of change had taken

place at the end of 24 hours, when the dilution exceeded 200 times the volume of the acid or potassium iodide solution. But in both series of trials, the figures conforming to no exact rule, they were repeated with the following more satisfactory results:

TITRATION OF 1 cc H_2SO_4—(1.845 Sp. Gr.)

	Sun-light.	Darkness.
With 200 cc H_2O.	0.67 cc	
" 150 " "	0.47	0.018 cc
" 100 " "	0.60	0.03
" 75 " "	0.37	0.06
" 50 " "		0.13
" 25 " "	0.33	0.26

The potassium iodide used in these and the following experiments was some especially prepared, and free from all impurities, except a trace of iodic acid. The amount was so small that none of the tests for iodic acid, except that with sulphurous acid, revealed its presence, and then only a scarcely recognizable indication. The same remark is true of the cadmium iodide, which was next tried, in order to learn whether the above laws were general in their application.

TITRATION OF 1 cc H_2SO_4—(With CdI_2).

	Sun-light.	Darkness.
With 200 cc H_2O.	1.15 cc	0.023 cc
" 150 " "	1.07	0.048
" 100 " "	1.09	0.03
" 50 " "	1 07	0.25
" 25 " "	0.95	0.13

On reviewing the methods pursued up to this point, three sources of serious error became apparent. 1st. The bottles, while placed in the sun-light during the day, were not titrated until the following morning, and during the intervening period, the law regulating the decomposition in darkness had the opportunity of operating, and that, too, in a contrary direction. 2d. The bottles were of various tints, some of yellowish or even pinkish tinge, which would affect the actinism of the solar ray very differently from quite colorless glass. 3rd. The bottles were of different shapes and dimensions, the latter circumstance causing the surface of exposure to increase in a very different ratio from that of the increase in bulk of the contained liquid only. For these reasons, comparison-tubes were substituted. In these, the increase in the surface of exposure of liquid would be directly proportional to the increase in its volume.

JAN. 18TH. SUNNED IN COMPARISON-TUBES (1 cc H_2SO_4).

	1st Series (A).	2d Series (B).
With 100 cc H_2O.	1.366 cc	1.366 cc
" 80 " "	1.83	1.40
" 60 " "	1.20	1.20
" 40 " "	1.00	1.00
" 20 " "	0.60	0.60

They were exposed from 12:20 P. M. to 2:45 P. M.

The changes of color were as follows, counting from beginning of experiment :

	TO VERY FAINT ROSE.	FAINT ROSE.	ROSE.	VIOLET.	BLUE.
100 cc A..	In 16 min.	In 19 min.	In 22 min.	In 30 min.	In 35 min.
100 " B..	8 "	14 "	17 "	25 "	31 "
80 " A..	10 "	16 "	21 "	28 "	33 "
80 " B..	13 "	17 "	23 "	28 "	33 "
60 " A..	15 "	20 "	26 "	28 "	34 "
60 " B..	19 "	19 "	25 "	28 "	34 "
40 " A..	21 "	25 "	27 "	30 "	35 "
40 " B..	20 "	25 "	27 "	29 "	35 "
20 " A..	24 "	27 "	32 "	35 "	40 "
20 " B..	22 "	25 "	30 "	32 "	38 "

From this and similar experiments, it would appear that the amount and rate of decomposition is proportional to the increase in the dilution, when this increases in the same ratio as that of the exposure to the sun's rays.

The next problem was, to determine the amount of decomposition in the presence of the same acid, when the iodine was united to different bases. Also, in the presence of different acids, when the iodine was united with the same base. The following solutions were therefore exposed from 10:45 A. M. to 4 P. M., to the sun's rays (Jan. 20th):

WITH DIFFERENT BASES.

20 cc H_2O + 1 cc KI + 5 cc starch	+1 cc H_2SO_4	= 6.5 mgrm. I.	CdI_2	= 6.5
40 " " + 1 " " + 5 " "	+1 " "	= 8.0 "	"	= 7.5
60 " " + 1 " " + 5 " "	+1 " "	= 9.3 "	"	= 9.5
80 " " + 1 " " + 5 " "	+1 " "	=10.5 "	"	=10.3
100 " " + 1 " " + 5 " "	+1 " "	= 9.5 "	"	=12.0

WITH DIFFERENT ACIDS.

20 cc	H_2O	+ 1 cc	H_2SO_4	+ 5 cc	starch	+1 cc	KI	= 6.5	mgrm.	I.		HCl	= 6.3
40 "	"	+1 "	"	+5 "	"	+1 "	"	= 7.8	"	"		=	7.5
60 "	"	+1 "	"	+5 "	"	+1 "	"	= 9.6	"	"		=	8.3
80 "	"	+1 "	"	+5 "	"	+1 "	"	=11.0	"	"		=	9.0
100 "	"	+1 "	"	+5 "	"	+1 "	"	=12.0	"	"		=	9.8

REPEATED, FROM 12 M. to 1 P. M.

100 cc	H_2O	+1 cc	KI	+5 cc	starch	+1 cc	H_2SO_4	=2.25	mgrm.	I.
100 "	"	+1 "	NH_4I	+5 "	"	+1 "	"	=2.20	"	
20 "	"	+1 "	KI	+5 "	"	+1 "	HNO_3	=0.90	"	
100 "	"	+1 "	"	+5 "	"	+1 "	"	=2.00	"	
20 "	"	+1 "	NH_4I	+5 "	"	+1 "	"	=1.00	"	
100 "	"	+1 "	"	+5 "	"	+1 "	"	=2.00	"	
20 "	"	+1 "	"	+5 "	"	+1 "	HCl	=0.30	"	
100 "	"	+1 "	"	+5 "	"	+1 "	"	=1.00	"	

In these trials, equal volumes of acid of usual strength were employed, not equivalent amounts of real acid, and the exact ratio of decomposition has yet to be determined.

Decomposition of Pure Acids in Sun-Light.—To determine whether the acids underwent decomposition in sun-light, nitric acid, of sp. gr. 1.4 ; hydrochloric acid, sp. gr. 1.202 ; sulphuric acid, sp. gr. 1.845, were taken ; 50 cc of each were placed in 100 cc glass-stoppered bottles, the stoppers luted in with paraffine, and the three bottles exposed at a south window, from Dec. 10th to Jan. 21st. At the end of this period, 1 cc of each was taken, diluted with 1 liter water, potassium iodide and starch-water added, and allowed to stand for 24 hours in darkness. They gave, on titration :

1 cc HNO_3 = 14.80 cc $Na_2S_2O_3$
1 " H_2SO_4 = none.
1 " HCl = "

To determine whether the *diluted acids*, with and without organic matter, changed by exposure to sunlight, 1 cc of each of the pure acids of densities given above, was diluted with 500 cc water. To three, starch-water was added. The six were sunned during six weeks, when potassium iodide and fresh starch-water added to each, and they were then titrated, after standing in darkness 24 hours. They gave :

	THE THREE WITH STARCH.	THE THREE WITHOUT.
500 cc dilute	HNO_3 = 0.50 cc	$Na_2S_2O_3$ = 0.30 cc $Na_2S_2O_3$.
500 " "	H_2SO_4 = none	= none.
500 " "	HCl = "	= "

The presence of the organic matter appears, in this experiment, to have facilitated the decomposition of the nitrate in sunlight.

Chemical Energy of the Solar Ray.—This investigation suggested a convenient method for studying the variations in the actinic force of the solar ray, which was to determine the amount of iodine set free by its action under identical conditions. In the following experiments, the first which were instituted in this direction, no attempt was made to determine absolute values. A series of comparison-tubes, each containing 1 cc H_2SO_4, 1 cc KI and 5 cc starch, with the amounts of water specified, were exposed at times and during intervals given, upon a comparator, the frame of which was kept normal to the solar ray.

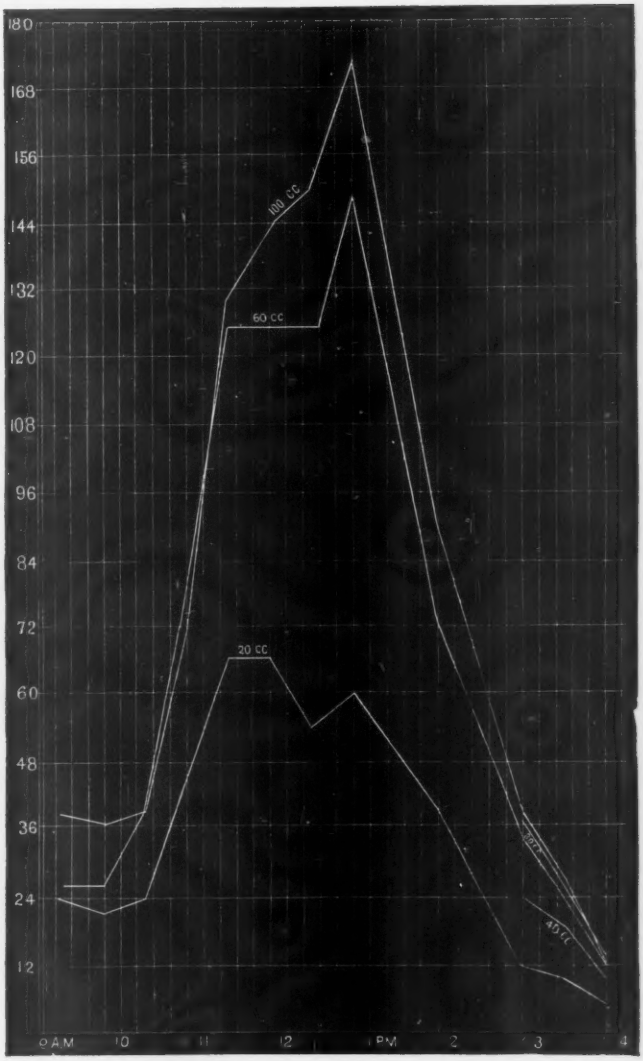
RELATIVE ACTION OF THE SUN'S RAYS (JAN. 22d, 1879).

	20 cc H_2O.	60 cc H_2O.	100 cc H_2O.
9 — 9:30 A.M....	0.24 mgrm. I.	0.27 mgrm.	0.38 mgrm.
9:30—10	0.22	0.29	0.37
10 —10:30	0.24	0.40	0.40
10:30—11	0.39	0.76	0.71
11 —11:30	0.67	1.25	1.29
11:30—12	0.67	1.25	1.46
12 —12:30 P.M....	0.55	1.25	1.54
12:30— 1	0.60	1.50	1.73
1 — 1:30	0.50	1.13	1.31
1:30— 2	0.40[clouded over]	0.75	0.92

CONTINUED, JAN. 24th.

	20 cc H_2O.	40 cc H_2O.	60 cc H_2O.	80 cc H_2O.	100 cc H_2O.
2:30—3	0.125	0.24	0.33	0.38	0.40
3 —3:30....	0.11	0.18	0.24	0.27	0.28
3:30—4	0.07	0.10	0.12	0.13	0.12

It will be seen by the inspection of the accompanying diagram, that the curve representing the amount of iodine set free in the 40 cc



CENTIMILLIGRAMMES OF IODINE.

HOURS IN THE DAY.

solution and the 80 cc solution, was intermediate between the curves of 20 and 60, and 60 and 100 respectively. Moreover, the determination of the liberated iodine is so easily made, that we possess in this method a convenient form of actinometry, lending itself readily to various practical applications. The chlorine-hydrogen method of Draper, which received at the hands of Bunsen and Roscoe, the various instrumental refinements essential to accurate quantitative results, requires a corresponding complexity of apparatus and nicety of manipulation, and is therefore carried into execution with difficulty by the working chemist. The method just described needs little or no apparatus beyond that essential to any well-equipped laboratory.

VI.—On the Influence of Variations of Temperature on the Deviation of Polarized Light by Solutions of Inverted Sugar.

By P. CASAMAJOR.

Received February 18, 1879.

The researches, of which I propose to give an account in this paper, were suggested by an interesting communication of Dr. Ricketts to this Society, which was presented at our last meeting.

Dr. Ricketts found that the temperature at which the deviation of a solution of inverted sugar becomes 0 is not 90° C., as given by some authors, but 92°, or rather 91°.7 C. From this Dr. Ricketts concluded, that if a solution of commercial sugar is inverted in the ordinary way, if, originally, the two constituents of the sugar were cane sugar and inverted sugar, after inversion there must only remain inverted sugar, and, if we bring this solution in the saccharometer tube to have the temperature of 92° C., the indication of the saccharometer scale must be 0.

As the inversion of sugar solutions in testing sugars is now almost entirely neglected, it struck me that the introduction of this subject before this Society was of great importance, as it is likely to excite inquiry in this direction, and must lead to interesting discussions which will throw much light on a ground which has not recently been explored. I hope the following remarks may be considered as a contribution to this important subject.

To enable me to present in a clear light, the results I have reached, it becomes necessary to bring before you some theoretical points relating to the inversion of sugar solutions.

It is to Mitscherlich that we owe the observation that the deviating power of solutions of inverted sugar on polarized light, varies with the temperature. It is, however, to Clerget that we owe a careful study of the subject. As far back as 1849, he published in the *Annales de Physique et de Chimie* (Vol. xxvi, 3d series, p. 175) a process for rectifying errors in tests of commercial sugars by Soleil's saccharometer, by taking into account the deviation caused by a solution of inverted sugar, obtained by heating with hydro-chloric acid a sugar solution, previously tested without inversion. In this communication, however, Clerget does not enter into theoretical considerations. He directs that sugar solutions shall be inverted by heating them with 10 per cent. of their volume of concentrated hydro-chloric acid, up to a temperature of 68° C., taking about 10 minutes in the operation. As soon as this temperature is reached, the solution is to be cooled down to some temperature between 10° and 35° C., as the table he gives for correction is calculated for temperatures between these limits, which, he says, "answer for all occasions which may present themselves in "Europe, as well as in the Colonies."

When Clerget proposed this new plan for making sugar analysis more correct, the subject of testing sugars was not a new one with him, for it is to him that we owe the idea of using Soleil's polarimeter as a special instrument for analyzing sugar, and, as far back as 1845, he had published, in the *Bulletin de la Societe d'Encouragement*, an account of Soleil's saccharometer. In his paper in the *Annales de Physique et de Chimie*, he gives no theoretical reasons for the new process. For the theory of the correction of the direct test of the saccharometer, by taking into account the test after inversion, I am indebted to some formulas in Mandlebluh's Guide (*Leitfaden zur Untersuchung der Verschiedenen Zuckerarten, Brunn*, 1867, p. 56). This theory I will now proceed to lay before you.

You are aware that commercial sugars contain, besides pure cane, sugar, a variety of substances, of which many exert deviating effects on a ray of polarized light. Among these is inverted sugar, which may be an immediate substance or a mixture of dextrose and lævulose. This latter supposition was advanced by Dubrunfaut, but has never been demonstrated in a satisfactory manner, although it would take volumes to collect what has been written about it, and is being written about it every day.

The other bodies which accompany cane sugar have not been studied in such a way as to throw light on their nature, with the exception of aconitic acid, isolated by Dr. Behr, who gave an interesting

account of his researches, in a paper read before this Society two years ago.

A point of great importance, in connection with the analysis of sugar by optical methods, is that the impurities which are generally present, are in such a condition that they seem to exert no effect on polarized light. The proof of this is to be found in the fact that with most sugars, particularly if the saccharometric test is above 90 per cent., the result, after the correction for inversion, is the same as given by the direct test. As many of the impurities reduce the alkaline solution of tartrate of copper, they are comprised under the head of *inactive glucose*, concerning the nature of which there are many unsatisfactory doctrines.

To explain how the direct test by the saccharometer may be corrected by subsequently inverting the solution on which this direct test was made, let us suppose that we have, in the first instance, a deviation, D. We may suppose that this deviation is the resultant of the following :

+ C, deviation due to the cane sugar.
— i " " " inverted sugar.
+ h " " " one or more substances, besides cane sugar, which turn the plane of polarization to the right.
— g, deviation due to one or more snbstances, besides inverted sugar, which turn the plane of polarization to the left.

Then we may suppose that $C - i + h - g = D$.

In this equation the only known quantity is D, but what we want to know is C. To find this quantity we make use of this fact, that if the solution under examination is heated with 10 per cent. of hydro-chloric acid, the whole of the cane sugar will be converted into inverted sugar, while the other substances will not suffer any change. From some experiments which I made with artificial mixtures analogous, as far as I could judge, to those which constitute the impurities of commercial sugars, I am led to believe that by heating with concentrated acid, a certain change takes place in the deviating power of the impurities of commercial sugars. This change is, however, very slight, and, as the impurities are in small quantities when compared to the cane sugar, the changes they suffer may be neglected, and we may suppose that, with the exception of the conversion of cane sugar into inverted sugar, no change takes place in the other constituents of the commercial sugar from the treatment with hydrochloric acid.

After this action of hydro-chloric acid, aided by heat, has taken

place, the solution is examined again in the saccharometer, and, after making a deduction for the volume of acid added, we will obtain a deviation d. This deviation, in ordinary sugars, will take place on the negative side of the scale. We may suppose that it is the resultant of the same elements that made up deviation D, with the exception of the quantity C, now represented by the deviation due to the inversion of the cane sugar originally present. This quantity we will call — I, and we shall have $-I - i + h - g = d$. If now we wish to eliminate the quantities i, h and g, we may easily do so by subtracting the second equation from the first, and we shall have: $C + I = D - d$.

By a series of experiments, Clerget established the relation between C and the corresponding value of I. This relation varies with the temperature, but we may suppose that the observation, after inversion, is taken at 28° C., at which a quantity of cane sugar which, before inversion, gives a deviation of 100 to the right, will show for the corresponding inverted sugar 30 to the left or — 30. At this temperature C + I becomes $1.3 \times C = D - d$. As d is a negative quantity, the algebraic subtraction is equivalent to an arithmetical addition, whence we draw the rule that at 28° C., the true quantity of sugar C, is equal to the sum of the two deviations divided by 1.3, as $C = \frac{D - d}{1.3}$ This number 1.3 corresponds, as we have said, to 28° C. We may, in the next place, inqnire: What are the corresponding numbers for other degrees of temperature? This brings us to the consideration of Clerget's table. For temperatures between 10° and 35° C., Clerget determined these numbers, and, on consulting his table, we find that, for a quantity of cane sugar equal to 100, we must take for the quantity D — d at 10° C., the sum 139, and that for every degree C. above 10°, up to 35°, the number representing the arithmetical sum of the deviations is equal to 139 minus one-half the difference between the number representing the temperature and 10°. Thus for 20°, we have $20 - 10 = 10$ and $139 - \frac{10}{2} = 134$; for 28°, we have $28 - 10 = 18$ and $139 - \frac{18}{2} = 130$; for 35°, we have $35 - 10 = 25$, and $139 - \frac{25}{2} = 126.5$, etc.

If the same law should hold good below 10°, we must, to 139, add $\frac{1}{2}$ for every degree below 10°, and this leads us to establish that at 0°, the number corresponding to D — d would be $139 + 5 = 144$. As this number 144 is one of great importance, allow me to recall to your attention, that it means that if we have a solution of pure sugar which will produce a deviation of 100 divisions on the positive side

of the saccharometer scale, if this solution is inverted by heating with 10 per cent. of hydro-chloric acid, this inverted solution, if tested at the temperature of 0° C., will, after making the correction for the quantity of acid added, produce a deviation of 44 on the negative side of the scale.

If we now suppose that the law which Clerget found for temperatures between 10° and 35°, holds good for all other temperatures, we may easily obtain the deviation to the left of the same solution of inverted sugar at any temperature, by subtracting from 44 one-half the number expressing this temperature in degrees Centigrade. Thus we shall have for 10°, $d = -(44 - 5) = -39$; for 20°, $d = -(44 - 10) = -34$; for 28°, $d = -(44 - 14) = -30$, and for any temperature t, $d = -(44 - \frac{t}{2})$.

If now, we draw a series of parallel equidistant lines, intersected by another series of lines, also parallel and equidistant, perpendicular to the first, we may take on the horizontal base line or line of the abscisses, a space between two lines corresponding to 1°C., and we may suppose that the space between two lines of the other series corresponds to a division of the negative side of the saccharometer scale. If now, on every vertical line, starting from the base line, we take a length proportional to the numbers given by the equation, $d = -(44 - \frac{t}{2})$, and if we connect the extreme points of all these lengths by a line, we will find that it is a right line, because the equation, $d = -(44 - \frac{t}{2}) = -44 + \frac{t}{2}$, is equivalent to the equation of the right line, $y = a + bx$, in which $d = y$, $a = -44$, $b = \frac{1}{2}$ and $x = t$.

If now, we take again our equation, $d = -44 + \frac{t}{2}$, we will find that, if the law it expresses holds good to the end, the temperature at which the deviation becomes 0 is 88° C.

The announcement that it was at 92° that d became equal to 0, led me to think, at first, that, possibly, the law that I had deduced from Clerget's table was not correct. It is true that the temperatures given by Clerget only extend from 10° to 35° C., but, between these limits, the law which governs the deviations of inverted sugar as affected by temperature, as deduced from his table, is represented by a right line. This I considered as an important fact, because I do not remember a single instance in which a law is expressed by a rigorous right line,

between limits that are sufficiently distant, in which this right line is not continued throughout. Having, then, very serious doubts about the accuracy of Clerget's table, I started to discover what the true law was.

For this purpose I prepared pure sugar, by taking the best cut loaf sugar and soaking it in 95 per cent. alcohol for several hours, taking the sugar out, letting the pieces drain, and drying them by hot air.

This sugar, tested before inversion, gave exactly 100 by the saccharometer. The solution, after the direct test, was inverted and tried again in the saccharometer at several degrees of temperature. After making a great many tests at various temperatures, I was forced to the conclusion that, not only is Clerget's table correct for temperatures between 10° and 35° C., but that the law represented by $d = -44 + \frac{t}{2}$ held good even beyond 88° C., at which temperature the deviation is equal to 0.

To enable an observer to seize at a glance the results I have obtained, I took a large sheet of cross-section paper divided very accurately into inches and tenths of an inch. On the line of the abscisses, I took for every degree Centigrade a space equal to $\frac{2}{10}$ of an inch, and, on the line of the ordinates, I took for every division on the negative scale of the saccharometer, $\frac{4}{10}$ of an inch. I then drew a right line, connecting the point representing — 44 of the Saccharometer scale with the point representing 88° Centigrade. This right line represents the law expressed by the formula $d = -44 + \frac{t}{2}$. Afterwards I plotted on this sheet 68 observations made at temperatures varying from 14° C. to 92° C. The result of this operation was that, out of 68 dots, 10 were entirely erratic, 18 were exactly on the line, and the remaining dots were so close that their distance from the line could be explained by an error of $\frac{1}{4}$ of a division in the observation of the saccharometer. In explanation of these results, I may state that the dots which I have called erratic were due to observations made on turbid solutions, or while the temperature was fluctuating. Observations made under such circumstances cannot be expected to be accurate. The other observations were made under conditions infinitely more trying to the eyes than those under which ordinary saccharometric tests are made. When the temperature fluctuates in the least during an observation, the disc of double quartz becomes distinctly elliptical, and, at times, the line of separation

between the two quartz plates swerves alternately to the right and to the left.*

To obtain the different temperatures required for these experiments, I had to alter a saccharometer so as to interpose a water bath between the two optical portions in a manner similar to that adopted by Dr. Ricketts. At the bottom of this water bath is an opening, communicating with the interior of a closed tube, three inches long, projecting at a right angle. To the closed end of this tube a Bunsen burner was applied when the water bath was to be heated. In this water bath I placed the tube containing the solution of inverted sugar. This tube is made of thin brass, and is closed at each end by a glass plate, held by a screw cap in the ordinary way. In the middle of this tube a portion was cut off, two inches long, and as wide as the diameter of the tube. Over the opening thus formed was soldered a projection, as shown in figures 1 and 2 at A. At first I used a tube with a

FIG. 1

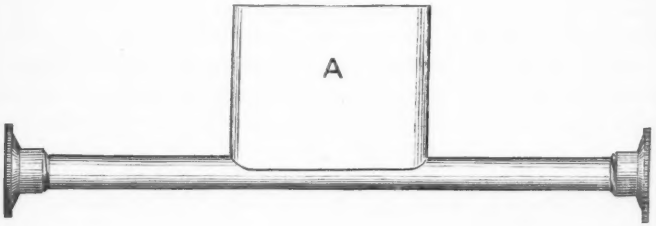
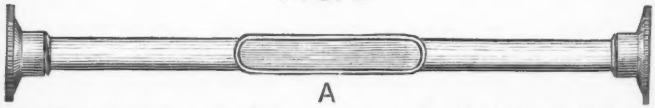


FIG. 2



cylindrical projection, of the same diameter as the tube itself, for the introduction of a thermometer to ascertain the temperature of the liquid in the tube, in the manner adopted for Clerget's thick glass tube. I think that the plan represented in fig. 1 and fig. 2 is preferable, as it allows the operator to agitate the solution by moving the thermometer backwards and forwards in the tube. A great advan-

*The sheet on which these dots are plotted was shown to the American Chemical Society. I have not thought it necessary to have it engraved, as the explanation given in the text makes the subject sufficiently clear.

tage obtained by having a projection of this shape is that the liquid in the projection does not rise or fall perceptibly when the thermometer is taken in or out. If the vertical projection has a small section, whenever a thermometer is placed in it the liquid rises in this projection, and a portion may be high enough to stand above the level of the water bath. Under these circumstances, this portion of the liquid gets cooler than the portion in the horizontal rube, and, if the bulb of the thermometer is longer than the diameter of the horizontal tube, the temperature indicated will be too low. Too much care cannot be taken in observing these minutiae, as otherwise accurate results cannot be obtained. Even with the greatest care, it is impossible to avoid errors in the observation of solutions whose temperature does not remain constant. In this connection I may be allowed to express the opinion that it was due to the want of a tube like the one represented in fig. 1 and fig. 2 that Dr. Ricketts was led to take 92° as the temperature at which d becomes equal to 0. I have repeatedly found at 92° that the deviation is on the positive side of the scale, ranging from 1.4 to 2, according as the observation was more or less accurately made. In his experiments, Dr. Ricketts did not take the temperature of the liquid in his tube, but that of his water bath. As his tube was made of glass, about one-eighth of an inch thick, we must suppose that the temperature of the liquid in the tube was different from that of the water bath, and that the difference between the two temperatures must depend on the rate of heating the water bath, *i. e.*, on the size of the flame.

I have mentioned that a thermometer with a long bulb may be the cause of serious errors; so may a thermometer whose movements are too sluggish. In the thermometer that I have used, the bulb was very short; the mercury column was very fine, so as to respond very quickly to slight variations of temperature. These thermometers have 40° C. on a scale six inches long. One goes from 0° to 40°, the next from 30° to 70°, and the third from 60° to 100°. Particular attention was paid in these experiments to keeping the water bath in continued agitation immediately before taking an optical observation, and while the observation was being made. The thermometer in the brass tube was also kept moving backwards and forwards for some time before looking through the tube. Unless these things are done, there is no certainty that the temperature observed is that of the liquid in the tube.

If, as I am convinced in this case, from the numerous experiments I have made, observations on solutions of inverted sugar, taken at any

temperature, are as reliable as those made at any other temperature, there can be no utility in heating the solution by the interposition of a water bath. Taking observations at the temperature at which the deviation is equal to 0, merely saves the trouble of dividing D — d by $144 - \frac{t}{2}$, and it is not certainly worth while to go out of our way for this purpose. The most convenient plan for making observations on solutions of inverted sugar is to follow the directions of Clerget, which are the following :

A sugar solution is placed in the ordinary way in the saccharometer, and the saccharometric test, D, is noted down. To a portion of this solution, say 50 cubic centimeters, are added 5 cubic centimeters of concentrated hydro-chloric acid. These are mixed by shaking up the graduated flask, and the flask is placed in a water bath and heated to 68° C., taking about 10 minutes in raising the temperature. This solution is afterwards immediately cooled to a temperature between 10° and 35° C. We then place it in a thick glass tube, provided with a vertical tubulure for the insertion of a thermometer to show the temperature of the liquid at the moment of observation. This tube is made 22 centimeters long, instead of 20 centimeters, so as to compensate for the addition of $\frac{1}{10}$ of hydro-chloric acid. If the temperature, at the time of the observation, is t, and the deviation to the negative side is d, we take the algebraic difference of the two readings, D — d, which is the arithmetical sum. To find the correct quantity of cane sugar C, from these observations, we must remember that for 100 per cent. of sugar, $D - d = 144 - \frac{t}{2}$, therefore, $\frac{C}{D-d} = \frac{100}{144 - \frac{t}{2}}$ and $C = \frac{(D - d) \times 100}{144 - \frac{t}{2}}$ *

*Since this paper was read before the American Chemical Society, Dr. Behr has kindly called my attention to a paper of Dr. Tuchschmid in Scheibler's *Zeitschrift* for 1870, p. 649. After a series of elaborate experiments, Dr. Tuchschmid concluded that Clerget's table was very reliable. He investigated the law of this table between 4° and 41°.8 C. Instead of the formula given above, he found $R = \frac{100\ S}{144.16035 - 0.50578\ T}$; R corresponds to C, S to D — d.

This formula leads to 87°.3 C. as the temperature at which the deviation becomes 0. The law of $d = -44.16035 + 0.50578\ T$ is also represented by a right line. As to the results given by this formula, as compared with the one I have given, they may be judged by the following :

Degrees C.	Tuchschmid.	Clerget.
10°	139.1	139
25°	131.747	131.5
35°	126.458	126.5
40°	123.875	124

If we have Clerget's table, we may, instead of making this calculation, follow the practical directions at the head of the table.

Instead of inverting by heating to 68° C., and then cooling down rapidly, it is advised by some authors that the solution be maintained at 70° for at at least 15 minutes. I can say, after doing both things repeatedly, that, in the second case, the solution is no better inverted than in the first. Some persons think it necessary to heat at 70° for an hour. This I did not try, as I had found no advantage in heating 20 minutes longer than Clerget directs.

For solutions that are to be heated above 68° for experimental purposes, it is necessary to neutralize the hydro-chloric acid by a base, as otherwise the solution becomes very red. For this purpose the preference should be given to carbonate of soda, which gives the best results. I put enough of carbonate of soda to make the solution slightly alkaline, and afterwards make it acid with a slight excess of acetic acid. In this condition the solution may very conveniently be placed in a brass tube. For some reason which I have not been able to discover, when the hydro-chloric acid used in inversion is neutralized by magnesia, the indications of the saccharometer are always too low.

When Venteke's saccharometer is used for testing inverted sugar, we are obliged to operate on solutions of greater dilution than the normal solution containing 26 gr. .048 of sugar for 100 cc, because the negative side of the scale is very limited, reaching only to — 16 on some instruments. A solution holding 26 gr. .048 of sugar in 100 cc is tested directly. Then 50 cc of this are transferred to a beaker ; the 50 cc flask is washed out, and the wash water is added to contents of beaker, and also 10 cc of hydro-chloric acid. After the solution has been inverted and saturated with carbonate of soda, if necessary, the whole is put in a 100 cc flask, and enough water is added so that the whole solution at 15° C. shall occupy 100 cc. The result of the test, after inversion, has then to be multiplied by 2.

I will conclude by giving from my books a series, 28 consecutive tests of raw and refined sugars. In the second column I give the direct test, before inversion ; in the third column, the correct test, as afforded by inversion. In a fourth column I have given the copper test for glucose, when I happened to have it. These glucose tests show that the substances which reduce the alkaline tartrate of copper are, for the most part, without action on polarized light.

Designation of Sugar.	Saccharometric Test (before inversion).	Corrected by Inversion.	Glucose, Copper Test.
Raw	86½	87	
"	85	86	
Refined—B	91½	92	
" —C	83	83	
Raw	77	80	
"	90½	90½	
"	91	90½	3.57
"	80	84	11.5
"	84	86½	
"	84	89	
"	75½	79	
"	79	81	
"	90	90	4.30
"	95	94½	
Refined	78	78	8.2
"	81	82	7
Raw	92	92	2.5
"	91	91	2.5
"	87	88	5
"	90½	91	2.5
"	88	87	
Manilla	82	81	7.25
"	84	82	7.92
"	83½	81	7.5
Melado	73½	74	7.5
Domestic	82½	81½	
"	85	86	
"	78½	76½	

In the case in which the test before inversion is lower than when corrected after inversion, the presence of an excess of lævo-rotary substance is indicated. When, on the contrary, the test, before inversion, is higher than after inversion, as in the case of the Manilla sugars, an excess of dextro-rotary substance is indicated. If these sugars are inverted, and the solutions of inverted sugar are tested in the saccharometer at 88° C., they would show deviations on the positive side of the scale. This, however, does not enable us to decide to what particular dextro-rotary substance the deviation is due.

VII.—Proceedings.

Regular Meeting, Thursday, March 6th, 1879, at 8 o'clock, P.M.

The meeting was called to order, Vice-President Leeds in the chair. In the absence of the Recording Secretary, Mr. Casamajor was appointed Recording Secretary *pro tem.* The minutes of the preceding meeting were read and adopted.

The minutes of the meeting of the Board of Directors, held at the office of Mr. W. H. Nichols, on Thursday, March 6, 1879, were then read.—*Present*—Messrs. Nichols, Endemann, Squibb, Casamajor, Leeds, Goldmark, Chandler, Waller and Goldschmidt. Dr. Squibb in the chair.

Dr. Waller reported that No. 4., Vol. 1, of the Proceedings, would be ready for the printer in about a week, a statement which was confirmed by Dr. Chandler.

The Treasurer reported $1,100 in the treasury.

It was moved and carried, that the publication of the Society should, in the future, be known as the "*Journal of the American Chemical Society*," which shall be published monthly, and contain the Proceedings, original papers, re-prints, *resumés* of special subjects, and abstracts of important papers published in other journals.

Moved and carried that the subject of admitting advertisements to the covers of the Journals be referred to the Committee on Publications, to report at the next meeting of the Board.

Moved and carried, that this Committee report a list of abstractors and reviewers of current chemical literature, and that the names of the Committee on Publications, the abstractors and reviewers, appear on the title page of the Journal.

It was moved that the sum of one hundred dollars be paid annually to the member of the Committee on Publications whom that Committee shall appoint working editor.

This motion was laid on the table.

The following gentlemen were then elected:

As Members, C. G. Wheeler, C. F. Erhardt, T. B. Stillmann, Aug. Eimer, W. F. Hildebrandt, J. F. Kelly; *Associates*, Joseph Binns, J. Seal, Wm. Ziegler, A. M. Despard.

The following names were proposed:

As Members, Wm. Van Slooten, Marcus Benjamin; *Associate*, John A. Scherer.

The first paper, entitled "Notes on Cupro-Manganese," by Augustus Raht, was read, the chairman remarking that an alloy composed of 7 parts copper, and 1 part of tin, easily cast, resists well the action of acetic acid and of moderately dilute sulphuric acid.

The second paper, "Notes upon Chicle," by Geo. A. Prochazka, Ph. D., and H. Endemann, Ph. D., was read; in the ensuing discussion, Mr. Stilwell said that he had found the coloring matter of chicle was easily soluble in water.

The third paper, by Prof. C. A. Goessmann, "The Examination of the Minnesota Early Amber Cane," was read by title and ordered to be published.

Dr. Ira Remsen gave a description of a new journal, devoted to chemistry, which he is about to publish.

Prof. Leeds then showed the Society an easy method for

THE DETECTION OF MINUTE TRACES OF WATER IN ALCOHOL.

Anthraquinone $C_{14}H_8O_2 = C_6H_4 <^{CO}_{CO}> C_6H_4$, is not only converted into hydro-anthraquinone $C_6H_4 <^{CO}_{CO}> C_6H_4$ by zinc dust and caustic soda, but also by treatment with sodium amalgam. When the hydro-anthraquinone so formed is brought into contact with water there is formed a clear, dark red solution of sodium-hydro-anthraquinone, which, on pouring off the excess of amalgam, gradually loses its color in contact with the air, or, if shaken, is quickly converted, needles of the anthraquinone separating. This reaction has been taken advantage of by Prof. A. Claus (Bee. d. Deutsch. Chem. Gesell. X, p. 927) to detect the minutest traces of water in ether, absolute alcohol, etc. If some crystals of sublimed anthraquinone are treated with absolute, alcohol-free ether, the crystals are converted into brownish-black crystals of sodium-anthraquinone. The least drop of water produces a magnificent red coloration, which disappears on agitation in contact with air, but reappears on standing. This can be repeated a dozen times, or as long as water and sodium-amalgam are still present.

When absolute alcohol is used, a dark green zone appears along the line of contact of amalgam and alcohol, which, on slight agitation colors the entire solution of a magnificent green, but entirely disappears on contact with air, anthraquinone being in all probability again formed.

Dr. Squibb said that anthraquinone was more sensitive to water than even anhydrous sulphate of copper. He also said that in the

manufacture of anhydrous alcohol, it should be distilled after its separation from lime, instead of while mixed with lime, as stated in the text books. Prof. Leeds also spoke upon the detection and estimation of nitrous acid by means of meta-diamidobenzol.

A letter was received from Dr. T. Sterry Hunt, accepting the presidency of the Society, and that on account of his absence in Europe, he had only lately learned of the honor conferred upon him.

Adjourned.

P. CASAMAJOR,
Recording Secretary pro tem.

The librarian reports that the following books and exchanges have been received by the Society:

ADDITIONS TO THE LIBRARY OF THE AMERICAN CHEMICAL SOCIETY.

From Jan. 1 to March 1, 1879.

PRESENTED BY AUTHOR.

Presented by Marcus Benjamin, Ph. B.:

"Dangerous Cosmetics." Pamphlet.

Presented by Gideon E. Moore, Ph. D.:

"On Chalcophanite: A New Mineral Species." Pamphlet.

"On the Chemical Constitution of the Wax of the Myrica Cerifera." Sheet.

"On the Electrolysis of the Substituted Derivatives of Acetic Acid." Sheet.

"On the Occurrence in Nature of Amorphous Mercuric Sulphide." Sheet.

"On Brushite." Pamphlet.

Presented by A. A. Hayes, M. D.:

"On the Wide Diffusion of Vanadium, and its Association with Phosphorus in many Rocks." Sheet.

Presented by John L. Hayes:

"Sheep Husbandry in the South."

Presented by Prof. Mariana Barcena:

"Vicente Fernandez, Analisis Cauylitativa Cuantitativa del Mezcal."

ACQUIRED BY GIFT.

Presented by S. A. Goldschmidt, Ph. D.:

J. Lawrence Smith; Paris Exposition (1867); "Report on Industrial Chemistry."

F. A. P. Barnard, LL. D.; Paris Exposition (1867); "The Industrial Arts and Exact Science."

"Society of Public Analysts." Proceedings, Vol. I, 1876.

Presented by P. Casamajor:

"Sugar Cane." Vol. X, 1878.

Presented by Waldron Shapleigh:

"Enquête sur la Question de l'Impot du Sucre a la Consommation." 2 vols.

Presented by Prof. Mariana Barcena:

J. W. Mallet. "La Barcenita" (a new mineral).

Presented by Messrs. Matthiesen & Wiechers:

"Kolbe's Journal fur Pracktische Chemie." Nos. 16 and 17, Vol. VIII (completing vol.).

Presented by Dr. C. F. Chandler:

"New York Board of Health." Report, 1874–5.

PURCHASED BY THE SOCIETY.

"Liebig, Kopp and Will, Strecker, Naumann, Fittica, Jahresbericht."

30 Vols; 1847–'76.
Part I, 1877.
Register, 2 Vols; 1847–'56, 1857–'66.
Part 1, Register; 1867–'76.

"Dingler's Polytechniches Journal."

3 Vols; 167, 168, 171.

No. 984 completing Vol. 170.

"Fresenius. Zeitschrift fur Anylytical Chemie."

14 Vols. and Register.

ACQUIRED BY EXCHANGE.

From Bureau of Statistics, Treasury Department.

Imports and Exports of the United States from July to Sept., 1875.

Statistics of Commerce and Navigation of the United States. 1877.

From the American Society of Civil Engineers.

Proceedings. 2 Vols; 1876–'7; Jan.—Sept., 1878, inclusive.

From the American Journal of Pharmacy.

"American Journal of Pharmacy;" 3 Vols; 48 (No. III lacking), 49 and 50.

From the American Philosophical Society.

Proceedings. 11 Vols.; 1861–'78.

From the New York Academy of Sciences.

Annals. Vol. I.

From the Newcastle-on-Tyne Chemical Society.

Transactions. Vol. III and first half of Vol. IV.

From the Italian Chemical Society.

"Gazetta Chimica Italiana;" 8 Vols.; 1871–'8.

APPARATUS.

Presented by Prof. A. R. Leeds.

Screen for Lantern.

List of Exchanges Received

During January and February, 1879.

AMERICAN.

Anthony's Photographic Bulletin.
The Pharmacist and Chemist.
" Journal of the Franklin Institute.
" Popular Science Monthly.
" American Journal of Pharmacy.
" Physician and Pharmacist.
" American Bookseller.
Van Nostrand's Engineering Magazine.
New Remedies.
The Philadelphia Photographer.
" Crockery and Glass Journal.
" Engineering and Mining Journal.
" American Gas-Light Journal.
" Oil, Paint and Drug Reporter.
" American Manufacturer and Iron World.
" Druggists' Circular.
" Brewers' Journal.
" Manufacturer and Builder.
" Mauufacturers' Review and Industrial Record.
" Brick, Pottery and Glass Journal.
" American Gardner.

ENGLISH.

The Chemical News.
" Journal of Applied Science.
" Chemical Review.
" Journal of the Society of Arts.
Sugar Cane.
Iron.

FRENCH.

Moniteur de la Teinture.
Revue des Industries Chimique et Agricoles.
Revue Industrielle.
Annales du Genie Civil.
Repertoire de Pharmacie.
Technologiste.

Journal des Fabricants de Sucre.
Moniteur Scientifique.
Moniteur Industriel.

GERMAN.

Berg und Huettenmaennische Zeitung.
Oesterreichische Zeitschrift fur Berg und Huettenwesen.
Polytechniches Notizblatt.
Photographische Mittheilungen.
Archiv der Pharmacie.
Deutsche Industrie Zeitung.
Wochenschrift fur Zuckerfabrickation.
Deutsche Gerberzeitung.

S. A. GOLDSCHMIDT,
Recording Secretary.

VIII.—Notes on Cupro-Manganese.

By A. RAHT, A. M.

Received March 6th, 1879.

Several French chemists have pointed out as early as 1869 that an admixture of manganese to copper, bronze and brass, tends to increase their hardness, elasticity and toughness. Since then, it appears that some parties in France have manufactured a compound styled "cupro-manganese," as a convenient form for alloying.

Some cupro-manganese was imported from France into this country about a year ago. Experiments have been made with it by large brass and bronze manufacturers; all these gave, however, negative results; the metal proving more apt to tear and crack under the rolls and punching-machines after an addition of this cupro-manganese.

No matter how much the inferior European copper may be improved by an admixture of manganese, one could hardly expect the same action on the superior quality of Lake Superior copper. However this may be, it is evident that even inferior copper could not be improved by the addition of a metal with such impurities, as shown by the following analysis of the imported French cupro-manganese.

It contains besides copper :

Manganese	16.86 %
Iron	0.91
Tungsten	0.20
Arsenic	0.19
Zinc	0.18
Lead	0.04
Nickel and Cobalt	0.05
Bismuth	Trace.
Antimony	Trace.
Phosphorus	Trace.

IX.—Examination of the Minnesota Early Amber Cane.

By Prof. C. A. GOESSMANN.

Received February 28, 1879.

The recent recommendation of the cultivation of the Minnesota early amber cane, an acclimated variety of the Chinese Sorghum, for the production of syrup and sugar for general home consumption, caused the investigation which I propose to describe shortly within the few subsequent pages. The entire management of the experiment, as far as the agricultural and industrial questions involved were concerned, was confined to the application of such modes of operation as any intelligent farmer could carry on with moderate means. The same apparatus were employed for the crushing and pressing of the cane, and the general treatment of the juice, which are quite extensively used for that purpose in Minnesota and other western states, *i. e.*, a Victor mill and a Cook's evaporator, with an additional sheet-iron pan for defecation.

Somewhat more than twenty acres, located on at least as many farms, in the vicinity of Amherst, were planted with genuine seed; upon the college grounds one acre was cultivated, which furnished mainly the material for my tests. As a correct appreciation of the circumstances which control the character of the final practical results, required a definite knowledge regarding the most favorable stage of the canes for sugar manufacture, a series of examinations were instituted with a view to ascertain that particular point. My thanks are due to Mr. E. B. Bragg for kind assistance in the earlier stage of the investigation.

The examination of the cane was carried out in the following manner: On the date specified, the stalks were cut off six inches above the ground, and two feet in length of the tops and the entire leaf

mass removed; the remaining part of the cane was subsequently crushed and pressed to secure its juice. The latter, after being tested for its specific gravity by Brix's saccharometer, and for its relative amount of free acid at boiling heat, by means of a solution of carbonate of soda, containing one grm. of sodium carbonate anhydride in 100 cc of dist. water, was treated without delay, with a solution of basic acetate of lead, in the same manner as in the case of the juice of the sugar-beet-root, to secure a good defecation for similar purposes. The filtered juice was subsequently divided in every instance into two portions; one part was treated directly in the usual manner, with Fehling's solution for grape sugar, and the other part, after being treated in the customary way with hydrochloric acid, at a moderate heat, to convert the cane sugar present into glucose, was treated like the former liquid for the total amount of sugar. The difference noticed between both tests was calculated according to well known rules as cane sugar. It has been the aim, during the entire investigation, to secure in all cases, not otherwise specified, a comparative value to the various analytical statements.

1.

1878.

Aug. 15.—Juice obtained from plants *five* feet high; no flower stalks in sight:

Specific gravity 4.2° Brix, at 27° C. temp.

Grape sugar present	2.48 per cent.
Cane sugar "	none
Soda solution required.....	6.8 cc.

The microscope revealed the presence of many granules of starch:

Cane lost at 100° C. 92.07 per cent. of moisture.
Cane left at 100° C. 7.93 " of solid matter.

2.

Aug. 16.—Juice obtained from plants *ten* feet high; no flower stalks in sight:

Spec. gravity 5.8° Brix, at 24° C. temp.

Grape sugar present..... ...	4.06 per cent.
Cane sugar "	none
Soda solution required.....	9. cc.

Cane lost at 100° C. 88.90 per cent. of moisture.
Cane left at 100° C. 11.10 " of solid matter.

3.

Aug. 20.—Juice obtained from plants with the lower leaves of the canes turned reddish; flower stalks well developed; flowers, however, not yet open:

Spec. gravity 7.0° Brix, at 24° C. temp.
Grape sugar present..... 3.47 per cent.
Cane " " ..... 2.15 "
Soda solution required..... 7. cc.
Cane lost at 100° C. 87.00 per cent. of moisture.
Cane left at 100° C. 13.00 " " solid matter.

4.

Aug. 24.—Juice obtained from plants bearing flower stalks with fully developed open blossoms:

Spec. gravity 8.7° Brix, at 23° C. temp.
Grape sugar present..... 3.7 per cent.
Cane " " ..... 3.0 "
Soda solution required ..... 4.0 cc.
Cane lost at 100° C. 85.93 per cent. of moisture.
Cane left at 100° C. 14.07 " " solid matter.

5.

Aug. 27.—Juice obtained from canes of plants in full blossom:

Spec. gravity at 10° Brix, at 25° C. temp.
Grape sugar present..... 3.65 per cent.
Cane " " ..... 4.13 "
Soda solution required ..... 10. cc.
Cane lost at 100° C. 84.52 per cent. of moisture.
Cane left at 100° C. 15.48 " " solid matter.

6.

Aug. 30.—Juice obtained from canes of plants with the formation of the seed fairly begun:

Spec. gravity 9.5° Brix, at 30° C. temp.
Grape sugar present ..... 4.00 per cent.
Cane " " ..... 3.81 "
Soda solution required..... 9.5 cc.
Cane lost at 100° C. 83.86 per cent. of moisture.
Cane left at 100° C. 16.14 " " solid matter.

7.

Sept. 2.—Juice from canes of plants with seeds in the milk, *i. e.*, seeds of full size, yet still soft :

Spec. gravity 10.70° Brix, at 27° C. temp.
Grape sugar present..... 3.85 per cent.
Cane " " ..... 4.41 "
Soda solution required ..... 9.5 cc.
Cane lost at 100° C. 84.15 per cent. of moisture.
Cane left at 100° C. 15.85 " " solid matter.

8.

Sept. 9.—Juice obtained from canes of plants with seeds still soft :

Spec. gravity 12.10° Brix, at 22° C. temp.
Grape sugar present..... 3.21 per cent.
Cane " " ..... 6.86 "
Soda solution required ..... 9.5 cc.
Cane lost at 100° C. 73.87 per cent. of moisture.
Cane left at 100° C. 26.13 " " solid matter.

9.

Sept. 9.—Juice obtained from canes of plants, from which, on the 2d of Sept., the leaves and the tops had been removed, without disturbing them otherwise :

Spec. gravity 12.8° Brix, at 22° C. temp.
Grape sugar present..... 3.77 per cent.
Cane " " ..... 6.81 "
Soda solution required..... 9.5 cc.
Cane lost at 100° C. 73.25 per cent. of moisture.
Cane left at 100° C. 26.75 " " solid matter.

10.

Sept. 18.—Juice obtained from canes of plants left upon the field without any alteration regarding leaves or tops :

Spec. gravity 13.20° Brix, at 22° C. temp.
Grape sugar present ..... 3.57 per cent.
Cane " " .... 7.65 "

11.

Sept. 18.—Juice obtained from canes of plants, from which only the tops had been removed, leaving the remaining portion of it undisturbed in the soil :

Spec. gravity 13.8° Brix, at 22° C. temp.
Grape sugar present..... 3.16 per cent.
Cane " " ..... 8.49 "

12.

Sept. 18.—Juice obtained from canes of plants, from which the tops and all the leaves had been removed on the 9th of Sept., whilst the remaining portion of it was not disturbed in the soil until cut on the 18th of Sept.:

Spec. gravity 11.5° Brix, at 22° C. temp.

Grape sugar present..... 3.16 per cent.
Cane " " ..... 5.85 "

13.

Sept. 18.—Juice obtained from canes of plants which were cut off on the 9th of Sept., had their tops removed as usual, yet their leaves left on, and, consequently been left upon the field for *nine* days before the sample tested was secured by pressing:

Spec. gravity 12.8° Brix, at 22° C. temp.

Grape sugar present..... 10.00 per cent.
Cane " " ..... 0.60 "

14.

Sept. 21.—The juice secured from the cane of No. 13 on the 21st of Sept., showed 13° Brix, at 21° C. temp., and when taken still two days later its specific gravity was equal to 15° Brix, at 18° C. temp.

From the previously stated observations, we may safely deduce the following conclusions, regarding the question above specified, at least as far as our climatical and terrestrial conditions bear on the growth and development of the Minnesota early amber cane, as a sugar-producing plant:

1. The grape sugar appears in the cane at an early stage of its growth (Nos. 1–2), and increases slowly to from 3 to 4 per cent. before cane sugar is formed.

2. The cane sugar is first noticeable at the time when the flower stalks become visible above the leaves, and its amount increases steadily until the seeds are of full size, yet still soft (Nos. 3–8).

3. The relative proportion of grape sugar to cane sugar did not exceed, at any time before the hardening of the seeds, 3.1 of the former to 8.4 of the latter; in the majority of cases it was about three to seven.

4. The cane loses a considerable amount of its moisture during the period of development of the seeds—from 10 to 12 per cent.; see Nos. 7–8; aiding thereby in increasing the density of the juice; the better quality of the latter during later periods in the life of the plant has, for this reason, to be ascribed largely to that cause, and not to the continued formation of sugar. The quality of the juice is improved at that stage largely, therefore, if not entirely, at the expense of its quantity.

5. The increase in the density of the juice of the cane, after the seeds are full sized, may be somewhat retarded by taking off the leaves, without disturbing the remaining plant in the soil (No. 12).

6. The cane sugar of the plants changes gradually, yet steadily, into grape sugar, after they are once cut off; the degree of that change varies widely, and depends largely on the exposure they suffer subsequently from weather and from temperature, being more serious during moist and warm, than in dry and cool weather.

7. The safest way to secure the full benefit of the early amber cane crop for syrup and sugar manufacture is to begin cutting the cane when the seed is full grown, yet still soft; in our case, between the 10th and 15th of September, and to grind them without delay.

The average yield of a syrup of 7.4° Brix, per acre, amounted to from 160 to 170 gallons, and rose in some instances to 240 gallons. To study the effect of the mode of manufacture pursued on the composition of the syrup, the following experiment was instituted. The juice of a healthy, fresh cut cane was tested before it passed into the defecator, and, also subsequently, the syrup obtained from it.

Sept. 29.—Juice 14.7° Brix, at 15° C. temperature.

Grape sugar present.....	3.61 per cent.
Cane " "	8.16 "

The syrup obtained from the previously stated juice contained:

Grape sugar.....	37.87 per cent.
Cane "	37.48 "

A glance at these results shows, that the relative proportion of the cane sugar and the grape sugar, as found in the juice of the same, are so seriously altered in the course of its manufacture into syrup, that it must be conceded, that the question, whether the syrup obtained in

the customary way can serve for the economical manufacture of sugar for the general market, can scarcely be entertained any more seriously; the sugar production, from syrup like the above, must remain a mere incidental feature in the amber cane industry, as far as our section of the country is concerned, and so long as the cost of separating the sugar does not offer more substantial advantages.

Amherst, Mass., Feb'y 26, 1879.

X.—Notes Upon Chicle.

By GEO. A. PROCHAZKA, Ph. D., and H. ENDEMANN, Ph. D.

Received March 19th, 1879.

The great interest in the search of substitutes for india-rubber and gutta percha, which for some time past has been manifested by technical men, has led us to an examination of a Mexican product, known in the United States for a number of years under the names of *Chicle* or *Sapota*. The latter name would imply that the product were derived from one of the many species of Sapotaceae, one of which is pointed out as the tree furnishing Balata. With the latter product it shares in fact many qualities; the general description given of Balata seeming to apply directly to the product under examination.

Balata is the concrete juice of a tree variously called by botantists *Mimusops Balata* Gaertn., *Achras balata*, *Achras Dissecta* and *Sapota Muelleri*, a Sapotacea which grows in British Guiana, while Chicle is said to be the product of a tree of the same class from Mexico. The difference in the manner of obtaining the material is evident from the chemical composition. While Balata is an almost pure hydro-carbon, with its various products of oxidation, Chicle contains, also, the various impurities of the juice from which it is derived.

The only reference to Chicle that could be found was by J. R. Jackson [Ph. J. Tr. (3) 7.409]. He gives a general description of the material, stating that it resembles gutta percha in appearance, being, however, more friable and brittle. He further mentions that it is probably derived from *Chrysophyllum glycyplacum* of the family Sapotaceae, and that it is also known under the names of *Mexican gum* and *rubber juice*.

In the course of the examination it became frequently necessary to consult the literature of caoutchouc, gutta percha and balata. In the following, we give the sources from which our information has been obtained.

On caoutchouc. Jahresber., 1847 and 1848. 742 ; Payen, Journ. pr. Ch., 55.273 and 56.196 ; S. Cloez and A. Girard, Jahresb., 1868. 494 ; J. Spiller, Jahresber., 1865. 576 ; A. Girard, Jahresber., 1868. 771 ; 1871. 800 ; 1873. 1127 ; Bouchardat, Jahresber., 1875.

On gutta percha. Soubeiran, Jahresber., 1847 and 1848. 743 ; E. N. Kent, Jahresber., 1847 and 1848. 744 ; Arppe, J. pr. Ch., 53.171 ; Payen, J. pr. Ch., 57. 152, and Jahresber., 1859 ; Bleekrode, Jahresber., 1859. 517 ; A. C. Oudemans, Jahresber., 1859. 517 ; E. H. von Baumhauer, J. p. Ch., 78. 277 ; A. W. Hofmann, Ann. Chem. Pharm., 115. 297.

On caoutchouc and gutta percha. Adriani, Jahresber., 1850. 519 and Chem. News, 2. 277, 289, 313 ; C. G. Williams, Jour. Chem. Soc., 15. 110, 121 ; A. W. Miller, J. pr. Ch., 97. 380 ; Berthellot, Jahresb., 1869. 334.

On balata. Bleekrode, Jahresb., 1859. 517 ; Sperlich, Wien. Akad. Ber., 59. (2) 107 ; O. Buchner, Dingl. Journ., 172. 146.

The material examined was in the shape of rectangular cakes, of light chocolate or flesh color, which was more pronounced on the surface, where atmospheric influences had acted more powerfully. The substance can be crumbled between the fingers ; it has, however, a certain degree of softness and tenacity, which is more perceptible after the material has been heated. Taken in the mouth it disintegrates, unites again, however, after chewing, then forming a soft, plastic mass. This latter quality has probably made it a favorite material for chewing-gum. On heating, it first evolves a sweet, caramel odor ; after this has disappeared the peculiar smell becomes perceptible, which is generated when caoutchouc or gutta percha are treated in like manner. The material disintegrates, if it is boiled with dilute acids ; the brown solution contains oxalic acid and saccharine matter. The residue subsequently boiled with dilute solutions of caustic alkalies unites again, and then forms a doughy mass.

The following constituents have been found :

Chicle resin or gum, forming 75 p. c. of the crude material ; oxalate of lime (with small quantities of sulphate and phosphate), 9 p. c. ; arabin, about 10 p. c. ; sugar, about 5 p. c. ; salts, soluble in water (chloride and sulphate of magnesia, small quantity of potash salts), 0.5 p. c. All these figures are, of course, only approximate.

Chicle Resin or Chicle Gum.—The resin, in the crude material, can be completely separated from the other constituents by bisulphide of carbon. If the finely divided material be shaken with about twice its weight of bisulphide of carbon, it will swell and assume a dark chocolate color. After standing for from two to three days, the insoluble residue will settle, leaving the yellow supernatant liquid perfectly clear. By carefully siphoning off the solution, and treating the residue with fresh portions of the solvent about ten times in the same manner, the resin will be completely removed from the insoluble residue, and obtained free from the other constituents of the crude product. Towards the end, when the solvent contains only little in solution, the insoluble residue will settle only very slowly. After distilling off the larger portion of the bisulphide of carbon, and pouring the residue into boiling water, the resin is obtained as a very light, fresh-colored, doughy, tolerably fluid mass, which is heavier than water. On cooling, it becomes hard, and then assumes a wax-like consistence, retaining, however, a certain degree of elasticity as long as it contains water or bisulphide of carbon. Dried at 100° C., it presents lumps, of somewhat granular appearance, which are light yellow inside, darker on the surface. They are brittle. Vulcanized at low temperature and with little sulphur, the resin becomes elastic, at higher temperatures and with more sulphur it becomes hard and brittle. The dried resin dissolves easily in bisulphide of carbon and cold ether, only partially in boiling alcohol. For reasons which will appear hereafter, the above method of obtaining the resin is objectionable, if its further examination is contemplated. Another method for obtaining the resin, at least free from arabin, the soluble salts, and the larger part of oxalate of lime, is by boiling the crude material with water. It then presents chocolate colored lumps, with properties similar to those described, in the resin, arrived at by the other method.

Arabin.—If the residue of the bisulphide of carbon treatment, or the crude material, is boiled with water for some time, milky, chocolate-colored liquids are obtained, which contain the arabin, saccharine matter and soluble salts in solution, and the oxalate of lime in fine suspension. A clear liquid can be obtained only by repeated filtrations. The clear solution is dark brown, almost opaque. In it the arabin can be separated from the other constituents, either by dialysis, or precipitation with four volumes of 97 p. c. alcohol. The flocculer precipitate formed is light, chocolate-brown. Dried at 100° it is dark-brown, and then contains 1.68 p. c. CaO, 1.68 p. c. MgO,

and 0.2 p. c. K_2O, and 0.34 p. c. of sulphate of lime. A purer material was obtained by re-dissolving the precipitate in water, acidulating the solution strongly by hydro-chloric acid, and effecting a partial precipitation by the addition of an equal volume of 97 p. c. alcohol. The larger quantity of the coloring matter remains in solution. The jelly, like transparent arabin precipitate, was washed by 97 p. c. alcohol; it became opaque and floccular, and by re-dissolving yielded a light-brown solution. This solution when kept hot may be almost completely decolorized by prolonged treatment with bone-black. Eventually a filtrate is obtained, which is turbid and gray by bone-black suspended in it. Addition of an equal bulk of 90 p. c. alcohol to the solution, will precipitate a portion of the arabin, together with the bone-black and the rest of the coloring matter. By filtration, a clear, light yellow filtrate is obtained, which, on repeated treatment by the method described by Neubauer, Ann. Chem. Phar., 102. 105, will yield arabin as a floccular, almost white precipitate. By dissolving this precipitate in water, evaporating the solution in the water bath, and drying the residue at 100° C., the arabin was obtained in the form of a light brown, very brittle glass, which shows all the properties generally ascribed to it. It is soluble in water.

A rough determination of the action on polarized light, gave a rotation to the left of about 37°.

Results of combustion in boat in current of oxygen:

I. 0.2530 gms. gave 0.3968 CO_2, 0.1384 H_2O, and 0.0015 ash.
II. 0.2388 " " 0.378 CO_2, 0.1335 " " 0.0012 "

making the per centage of C and H, after the deduction of the ash,

	I.	II.	$C_6H_{10}O_5$ Calculated.	$C_{12}H_{22}O_{11}$ Calculated.
C.	43.03	43.39	44.44	42.10.
H.	6.11	6.24	6.17	6.43.
O.	50.86	50.37	49.39	51.47.
	100.00	100.00	100.00	100.00.

The substance analyzed may be looked upon as a mixture of the two compounds $C_6H_{10}O_5$ and $C_{12}H_{22}O_{11}$. The filtrate of the precipitate that had been the source of the above mentioned purer material, yielded a further quantity of arabin, on addition of an excess of alcohol, in the form of a chocolate-colored powder, which, after drying at 100°, was insoluble in water. It dissolved, however, on the addition of a small quantity of alkali. The residual alcoholic liquid

contained the bases as chlorides, produced by the addition of hydrochloric acid, and coloring matter; the latter separates in the form of a dark-brown powder, on removal of the alcohol by distillation.

Saccharine Matter and Soluble Salts.—It has been stated above, that arabin can be separated from the other constituents of the original aqueous solution, either by dialysis or by precipitation with alcohol. In the first case, the sugar and the salts are found in the crystalloid liquids, in the latter they are contained in the alcoholic solution. From the latter solution they are obtained in a purer condition by dialysis. The solutions, on concentration, will yield liquids of caramel odor and color, and bitter sweetish taste. On evaporation to dryness, brown, hard, transparent residues are obtained. The aqueous solution reduces Fehling's solution. Attempts in different directions to further the character of the sugar were unsuccessful. No definite product could be obtained, owing, perhaps, to the large proportion of salts, which form one-tenth of the total quantity of solids in solution. The sugar may possibly be arabinose, derived from the arabin, in the alteration of the raw material.

The soluble salts are inorganic, being mainly chloride and sulphate of magnesium, potassium salts and sulphate of lime.

Oxalate of Lime is easily identified. It remains in the form of a light brown, very fine powder, which runs through the filter easily, on filtration of the aqueous extracts mentioned under arabin (p. 52). By dissolving it in hydrochloric acid and reprecipitating by neutralization with ammonia or carbonate of soda, added till the liquid was only very slightly acid, the oxalate of lime was obtained in the form of a slightly gray powder.

A portion of this was dried at 100°, and then ignited; 0.7972 gms. gave CaO 0.3127 or 39.2 p. c.

CaC_2O_4 + aq contains CaO 38.36 p. c.

The discrepancy is easily explained, as the material examined, contained small quantities of sulphate and phosphate of lime, and silica. Oxalic acid in its pure crystallized form, and various oxalates, were also prepared.

EXAMINATION OF THE CHICLE RESIN.

An examination of the gum, as obtained by either of the methods, above mentioned, showed it to consist notably of four different compounds. All of these, when heated, show a similarity in their behaviour; they evolve a peculiar, not disagreeable gutta odor, become soft and transparent, increasingly so as the temperature rises. A distinct melting point, therefore, cannot be assigned to them.

Two of the compounds, forming 75 p. c. of the resin, contain oxygen and correspond in their formulas, $C_{10}H_{16}O$ and $C_{20}H_{32}O$, as well as in their properties, to those described by Payen and Oudemans, as obtained from gutta percha, and termed by the former, alban and fluavil.

The first of these oxygen compounds, while being only slightly soluble in cold water, dissolves easily and in large quantity, on boiling; the latter dissolves easily in cold alcohol.

The other two constituents of the resin are hydrocarbons, $C_{10}H_{16}$. They are insoluble in boiling alcohol.

By protracted action of boiling alcohol on the chicle gum, only the oxygen compounds go in solution, while the hydrocarbons remain undissolved. A ready means for separation is thus offered.

Oxygen Compounds.—If an excess of 94 p. c. (vol.) or even more dilute alcohol is added to the chicle gum, and its temperature raised to the boiling point, and maintained at or near this point for several hours, it assumes a yellow color. The liquid obtained by decanting, or rapidly filtering off the boiling yellow solution, on cooling, soon becomes turbid, and after standing 24 hours an ample, verrucose crystallization will be deposited both on the bottom, and in more distinct crystals on the sides of the vessel, containing the solution. The supernatant liquid was light yellow and perfectly clear.

Chicle Alban.—$C_{10}H_{16}O$. The warts, measuring from 1 to 2 mm in diameter, were either obtained of a milky opalescence or pure white and opaque. Dried at the air and over sulphuric acid, they all become perfectly white and opaque. Some of the opalescent crystals were carefully selected from the sides of the vessel, thrown on a filter, washed repeatedly with 94 p. c. alcohol, dried over sulphuric acid, and reduced to a fine powder, which was first dried again over sulphuric acid, and eventually at 100°. Thus a light yellow powder was obtained, which baked together slightly at the temperature at which the drying was performed.

Combustion yielded the following results:

I. 0.305 gms. gave 0.8925 CO_2 and 0.2995 gms. H_2O.
II. 0.2601 " " 0.762 " " 0.2525 " "

	I.	II.
C.....	79.81 p. c.	79.90 p. c.
H.....	10.91 "	10.78 "
O.....	9.28 "	9.32 "
	100.00	100.00

These analyses do not represent the pure alban.

The bulk of the substance prepared for analysis was, therefore, redissolved in a small quantity of 94 p. c. (vol.) alcohol, by the aid of heat. On cooling, the whole liquid gelatinized. The gelatinous precipitate, which resembles gelatinous silica in appearance, was thrown on a filter, washed thoroughly by cold alcohol, re-dissolved, re-crystallized, and thus obtained pure. The pure substance, after drying over sulphuric acid, presents a perfectly white, very bulky powder. 100 pts. (wt.) of watery alcohol (0.82 sp. gr. at 13°), dissolve of it 0.66 pts. at 14°, and 0.71 at 17°. Boiling alcohol dissolves it easily, and in considerable quantity, and it is deposited, on cooling, either as a gelatinous mass or in the form of milky opalescent or white warts. It is very easily soluble in ether, chloroform, naphtha, benzol, and bisulphide of carbon.

When heated, it bakes together slightly at 100°, without changing its external appearance ; at 106° it unites in a transparent mass, becoming softer as the temperature rises ; at 145° it forms a syruppy light brown mass, which, on cooling, solidifies to a light brown transparent, very brittle glass.

A portion of it, heated in a boat in a current of dry carbon dioxide to 100°, unto constant weight, on combustion, yielded the following result :

III.—0.2351 gms. gave 0.6810 CO_2 and 0.2148 H_2O, and no ash.

	III.	$C_{10}H_{16}O$. Calculated.
C.....	79.00 per cent.	78.96 per cent.
H.....	10.15 "	10.52 "
O.....	10.85 "	10.52 "

The result of the analysis corresponds closely to the empiric formula, $C_{10}H_{16}O$.

Chicle Fluavil ($C_{20}H_{32}O$?).—The substance represented in the analyses Iand II, may be looked upon as a mixture of between 2.5 to 3 pts. of pure alban, with 1 pt. of a lower oxygen compound, $C_{20}H_{32}O$ (fluavil ?). This assumption is further confirmed by the determination of the total quantity of solids, in the solution, obtained by digesting the substance for some time with a small amount of alcohol. The figures arrived at, are much higher than would correspond to the solubility of pure alban, as represented by analysis III, in alcohol of the same strength. Thus 0.953 gms. digested for some time with 25 gms. of alcohol (0.82 sp. gr.), gave a solution which at 17° contained 1.57 pts. of resin to 100 pts. of alcohol (0.82 sp. gr.). Assuming that the whole quantity of fluavil, contained in the substance, has gone in solution, together with as much alban as corresponds to its solubility in a pure

condition, a similar quantitative ratio, in regard to the supposed constituents, is reached.

The application of the difference in solubility in alcohol to the separation of alban and fluavil, while it admits the preparation of the former in a perfectly pure condition, will yield the latter only in a very approximate state of purity.

The clear, light yellow, supernatant liquid of the alban crystallization, mentioned in an earlier part of this paper, on concentration and standing, will give an additional yield of alban, less pure, however, than the first. The mother-liquor, thus obtained, on concentrating, cooling and standing, will deposit a slightly yellow, translucent, viscid mass, which contains a large proportion of alcohol. The deposit melts at a very low temperature, spreads and covers the plate, to which it was transferred, in an even, thin layer. By protracted heating most of the alcohol can be removed. On cooling, the substance then presents a transparent, brittle glass, of light lemon-yellow color.

The last traces of alcohol offer some difficulty in their removal. A portion of the glassy mass was reduced to a very fine powder; this was very thoroughly mixed, spread out in a very thin layer on a plate, dried at ordinary temperature for some time, and finally for a number of days at 100°. The powder unites and melts much below this temperature. The mass on cooling solidifies again to a light yellow, brittle glass. Pieces of this were directly used for analysis. The combustion was performed in oxygen. Constant results, however, could not be obtained.

I. 0.2462 gms. gave 0.739 CO_2 and 0.2465 H_2O; no ash.
II. 0.2483 " " 0.7333 " " 0.2403 " "
III. 0.2579 " " 0.7565 " " 0.245 " "
IV. 0.2866 " " 0.8335 " " 0.2710 " "

	I.	II.	III.	IV.	$C_{20}H_{32}O$. Calculated.
C.	81.86 p. c.	80.54 p. c.	80.00 p. c.	79.32 p. c.	83.33 p. c.
H.	11.13 "	10.75 "	10.55 "	10.51 "	11.11 "
O.	7.01 "	8.71 "	9.45 "	10.17 "	5.56 "
	100.00	100.00	100.00	100.00	100.00.

The glassy mass dissolves easily and completely in boiling alcohol. The light, yellow solution on cooling and standing deposits, besides amorphous viscid fluavil, a very ample verrucose crystallization of alban. By the latter fact, and by the above quoted analyses, it would appear that fluavil were capable of further oxidation to alban.

It is to be presumed that the analysis would have yielded a lower per centage of oxygen, if the substance had, at once, after obtaining it, been dried in a current of dry carbondioxide. The compound, however, possesses only few very characteristic properties, and for the present, at least, does not appear of sufficient interest to warrant any further attempts at obtaining it in a perfectly pure condition.

100 pts. (wt.) of alcohol of (0.82 sp. gr.) dissolve at 13.5°, 2.6 pts. of fluavil.

Hydrocarbons $C_{10}H_{16}$.—By long and repeated digestions of the chicle gum with fresh quantities of alcohol, the larger portion of the oxygen compounds may be removed; ultimately, a tolerably liquid residue is obtained, which resists the further action of the solvent. On cooling, this residue assumes a more wax-like consistence. It is of light gray color, if prepared from the resin, obtained by bisulphide of carbon. The gum, resulting from boiling the crude material with water, yields it in the form of a dark, chocolate-colored mass, which contains oxalate of lime. In both cases the residue consists mainly of two hydrocarbons of the empiric formula $C_{10}H_{16}$, and alban and fluavil which boiling alcohol has failed to remove.

The difference in the behavior of the two hydrocarbons to the solvent action of ether, offers a ready means for their separation. One of them, while it is altogether insoluble in cold ether, dissolves easily and in very considerable quantity, on boiling. From this solution it separates completely, and in a crystalline form, on cooling. The other hydrocarbon seems to be amorphous, and is readily soluble in cold ether.

The complete separation of the hydrocarbons from the oxygen compounds, presents some difficulty. The hydrocarbons themselves oxidize very easily. Although they were not obtained in a perfectly pure condition, owing principally to the smallness of their quantity in the raw material, yet certain features in behavior, alike prominent in their different stages of purity, and the steady decrease of the per centage of oxygen in the course of their purification, allowed definite conclusions to be drawn, as to their properties and composition in a pure state.

Hydrocarbon $C_{10}H_{16}$, *soluble in cold ether.*—If the previously mentioned chocolate-colored residue, from which most of the alban and fluavil had been removed by repeated treatment with boiling alcohol, is digested for some time with an excess of boiling ether, the hydrocarbons and remaining oxygen compounds will completely

dissolve ; the liquid obtained is, however, turbid by suspended oxalate of lime and coloring matter. Upon standing for 24 hours at very low temperature the insoluble hydrocarbon will have separated completely in a crystalline form ; the clear light yellow liquid then obtained by filtration, yields no further crystallization on concentration.

In the manner indicated, a solution was arrived at which contained only the soluble hydrocarbon, besides alban and fluavil. This, after concentration, was poured into about four times its bulk of 94 p. c. (vol.) alcohol. A white partly powdery and partly flaky precipitate forms. The mixed liquids were at once heated and distilled to about two-thirds of their original bulk. In this operation, most of the precipitate unites in a light flesh-colored, doughy mass, which attaches itself to the bottom of the vessel ; a portion, however, remains finely suspended in the liquid, giving it a milky appearance.

After the liquid had been distilled to the required bulk, it was decanted off, boiling. The residue was subjected to repeated and prolonged treatment with fresh portions of boiling alcohol, which were always decanted off, boiling. Thus, eventually, a light flesh-colored, soft and very tenacious mass was obtained. A portion of this was dried at 100° in a dry current of carbondioxide. On combustion the following results were obtained :

0.2365 gms. gave 0.7378 CO_2 and 0.2437 H_2O ; no ash.

C=85.08 p. c. H=11.45 p. c. O=3.47 p. c.

The remaining portion was dissolved in ether, and the solution subjected again to the previously described treatment with alcohol.

The same operations (redissolving in ether, etc.) were repeated with the residue obtained. The substance eventually obtained was not very different, in its general properties, from the one represented in the above analysis. It was soft, very tenacious, of light gray color and somewhat translucent. Its total yield was about one-tenth of the original portion. A quantity prepared for analysis in the same manner as before, on combustion, gave the following results :

0.2967 gms. gave 0.9435 CO_2 and 0.3043 H_2O ; ash visible, has no appreciable wt.

		$C_{10}H_{16}$. Calculated.
C	86.73	88.23
H	11.40	11.77
O	1.87	

Further purification, without doubt, would have eventually yielded the pure hydrocarbon. This, however, had to be abandoned in consequence of the small yield of the substance from the raw material,

still further reduced by the various above mentioned operations. The two analyses given, the constant ratio of carbon and hydrogen in both, allows the inference of the empiric formula $C_{10}H_{16}$ for the pure hydrocarbon. The constant properties of the substance in its different stages of purity, allow the assumption of these same properties for the hydrocarbon in a pure condition.

The principal properties have already been given. The substance is soft, very tenacious and translucent at ordinary temperature. When boiled with alcohol it becomes softer, but retains its tenacity. On heating it becomes softer, but even at 135° it is still gelatinous. It dissolves very easily in naphtha, chloroform and bisulphide of carbon. Cold ether first converts it into a limpid, perfectly colorless mass, which it is very difficult to distinguish in glass vessels, and then rapidly dissolves it. Warm ether dissolves it even more rapidly. From these solutions it could not be obtained in a crystallized or powdery form.

Hydrocarbon $C_{10}H_{16}$, *insoluble in cold ether.*—The crystalline residue of the solution that had originally yielded the hydrocarbon, contains the entire quantity of the insoluble compound, together with small quantities of oxalate of lime and coloring matter. This residue was thoroughly washed by cold ether and pressed between blotting paper. By re-dissolving in warm ether and filtration, oxalate of lime and coloring matter were completely removed. A solution was thus obtained, which was perfectly colorless and clear, when warm. The filtration of the boiling liquid presents some difficulty; as the ether evaporates, the crystallization begins already on the filter, thus retarding or altogether interrupting further filtration.

The clear, colorless liquid crystallizes on cooling; after standing 24 hours it has become one mass of white microscopic crystals. These were thrown on a filter, thoroughly washed by cold ether and pressed between filtering paper. A portion of the substance thus obtained was dried in the air bath at 110°; it melts, becomes transparent, and on cooling presents a light brown, not a very homogeneous mass of a consistence, somewhat harder than wax. Pieces of this mass were transferred to a boat and subjected to combustion. The following results were obtained:

I. 0.3022 gms. gave 0.9215 CO_2 and 0.29 H_2O; 0.0004 white, amorphous ash.
II. 0.3036 " " 0.934 " " —— " 0.0006 white, " "

Calculated percentages after the deduction of the ash:

I. C=83.27 p. c. H=10.68 p. c. O=6.05 p. c.
II. C=84.07 "

The bulk of the brown mass was re-dissolved in boiling ether. A portion of the white crystalline powder obtained from the filtrate on cooling was at once transferred to a boat and dried at 100° in a dry current of carbon dioxide. On analysis, the following results were obtained :

III. 0.3030 gms. gave 0.93 g. CO_2 and 0.2955 H_2O ; no ash.

C=83.55 p. c. H=10.81 p. c. O=5.64 p. c.

The rest of the crystalline powder, when re-dissolved in boiling ether, re-crystrallized and otherwise treated in the same manner as before, on combustion, yielded the following results :

IV. 0.3148 gms. gave 0.9773 gs. CO_2 and 0.3108 H_2O ; no ash.

C=84.67 p. c. H=10.97 p. c. O=4.36 p. c.

The remaining bulk of the substance, obtained from the original etheric solution, was re-dissolved in ether, and an equal bulk of 94 p. c. alcohol added. A white powdery precipitate forms, which unites to a white liquid mass ; a portion of it, however, remains in milky emulsion, even after long continued boiling. The subsequent treatment is essentially a repetition of the method applied for the purification of the soluble hydrocarbon. The residue eventually obtained, on cooling, presents a white brittle mass, which is harder than wax. This was digested for a number of days with renewed portions of ether. It swells, disintegrates to white floccular masses, but does not dissolve. These floccular masses were heated again with alcohol. They unite, melt, and on cooling are obtained in the original white wax-like brittle form. A portion of the substance thus obtained was dried in a current of carbon dioxide. Combustion yielded the following results :

IV. 0.2043 gms. gave 0.6459 CO_2 and 0.2123 H_2O ; 0.0003 ash. Calculated after deduction of the ash :

C=86.37 p. c. H=11.57 p. c. O=2.06 p. c.

The bulk of the substance was again subjected to the same purifying method, with the only exception that the floccular masses obtained by digestion with cold ether, were at once dried in a current of dry carbon dioxide. The results of the analysis were as follows :

V. 0.2061 gms. gave 0.6560 CO_2 and 0.2152 H_2O ; 0.0002 ash.

		$C_{10}H_{16}$. Calculated.	
C.....	86.89	C.....	88.23
H.....	11.61	H.....	11.77
O.....	1.50		

The results are not materially different, if the drying has been performed in hydrogen.

What has been said about the further purification of the soluble hydrocarbon, might be justly repeated here. The analyses permit the same inferences to be drawn. In the present case the purification is even more difficult. The substance is very oxydizable. An analysis of the same material represented in analysis V, which was made on the day following, yielded the following results :

0.3689 gms. gave 1.158 CO_2 and 0.3742 H_2O ; 0.001 ash, or,

C = 85.84 p. c. H = 11.3 p. c. O = 2.86 p. c.

In this case the drying of the substance was done in hydrogen. The analysis shows that it is absolutely necessary to perform the analysis directly after obtaining the substance. The substance approaches very closely, or is, perhaps, identical with the gutta examined, and obtained from gutta percha by V. Baumhauer. The crystalline substance, as obtained from the warm ether solution, on cooling, presents a white powder, on drying at ordinary temperature ; in this form it oxydizes even more rapidly. The air in the bottles in which it was kept, contained ozone in considerable quantity. Occasionally the hydrocarbon was also obtained in the form of white verrucose masses, about 0.2 mm. in diameter. The substance represented in analysis V, is a white, slightly translucent mass, which is brittle at ordinary temperature. At 45° it becomes soft and transparent, and evolves a very strong and agreeable gutta odor ; it is syruppy at 125°. At no temperature does it show the tenacity, peculiar to the soluble hydro-carbon. It dissolves easily in benzole and chloroform. In cold ether and naphtha it swells, and dissolves on boiling, and is deposited again in a crystalline form, on cooling.

It may not be amiss, in this place, to mention again that the chicle resin dissolves easily in cold ether, which shows that the hydro-carbon is readily soluble in cold ether in the presence of an excess of the oxygen compounds.

Vulcanized products.—The chicle resin, obtained by bisulphide of carbon, yields, on exhaustive treatment with boiling alcohol, a light gray residue (p. 58) which dissolves readily on digestion with boiling ether. Only a very slight floccular precipitate remains. The clear light yellow solution thus obtained, gelatinizes to a mass of white crystals upon cooling and standing. Evaporation of the mother liquid, obtained by filtration, yielded a residue which, on drying at 100°, presented a light brown, slightly transparent elastic mass. This residue was almost utterly insoluble in ether, naphtha, bisulphide of carbon and chloroform, even on boiling. It becomes gelatinous, translucent and light yellow on digestion with ether, and

swells to about twenty times its original bulk, and only very little is dissolved. This peculiar behavior at once suggests that the residue may be a vulcanized product which was fully confirmed, on further examination. A portion of the substance, which had been repeatedly treated with boiling ether and alcohol, and then dried at 100°, proved to contain 1.77 p. c. of sulphur and about 8 p. c. of oxygen.

The waxy mass obtained by drying the previously mentioned white crystals at 100°, after they had been thoroughly washed by cold ether and pressed between blotting paper, consists largely of the insoluble hydro-carbon, but contains also a similar vulcanized product which remains behind undissolved on digestion with boiling ether.

The source of the sulphur in the vulcanized products is readily comprehensible. It is a well-known fact that bisulphide of carbon will alter on standing, exposure to light and air, and then contain free sulphur. (Compare, also, Gmelin, Handbuch der anorg. Ch., 6te Aufl., Vol. I. (2), p. 224.) The complete extraction of the chicle resin from the raw material is naturally a lengthy operation, and atmospheric influences on the bisulphide, have ample time to exert themselves. Hence the hydro-carbons were obtained first contaminated with sulphur, and finally in a vulcanized condition.

The descriptions given of gutta are somewhat conflicting, and it has been assumed that gutta percha may exist in different modifications. The presence of these two modifications, in the same sample, has never been proven.

The properties given to gutta by various authors are such, that they seem to describe as gutta, either a hydro-carbon soluble in cold ether, or a hydro-carbon soluble in warm, but insoluble in cold ether.

From the manner in which these investigations were made, it remains doubtful whether the authors, after finding one, really looked for another hydro-carbon.

The hydro-carbons found by us in chicle correspond, in many respects, to these different guttas, and must be classed in the same group with them, if they are not actually identical—a fact, the proof of which would have necessitated a direct, comparative examination of guttas from various sources.

Similar comparative examinations are also necessary to establish the identity or difference of the alban and fluavil found by us, with or from those described by others.

While we found the melting point of chicle alban at about 145° C., Oudemans gives the melting point of gutta percha alban at 140° C., and Payen at 160° C.

The authors who have described and examined gutta percha, have looked upon the compounds containing oxygen as secondarily derived from the hydro-carbons, and have assumed that the original juice contained only these. From the properties of the hydro-carbons and from observations made by us, we have come to similar conclusions, as regards chicle.

The chicle resin which we have examined, contains the above described constituents in about the following proportions :

Alban	45 per cent.
Fluavil	30 "
Insoluble hydro	17 "
Soluble carbon	8 "

From the above, it is evident that but little of the original hydro-carbon has escaped oxidation ; and the difference in the properties of the resin from the properties of the original hydro-carbon, is readily comprehensible.

The crude chicle contains besides, 75 per cent. of chicle resin of the above given composition :

Arabin	10 per cent.
Sugar	5 "
Soluble inorganic salts	0.5 "
Oxalate of lime (sulphate and phosphate)	9 "

From this it is evident that chicle is merely the product of direct evaporation of the juice, without attempt of separation, as is practised in the case of gutta percha and india rubber.

There is no doubt in our minds, that by proper treatment of the raw juice a far more valuable product would be obtained than the chicle gum which is now in the market.

Whether the product, then obtained, will be one similar to gutta percha, balata or india rubber, must be left to future examination of the raw juice, which so far we have been unable to obtain.

XI.—Influence of Light upon the Decomposition of Iodides.

By ALBERT R. LEEDS.

Received March 31, 1879.

As long ago as 1845, a sharp controversy arose between Schönbein and Prof. Fischer, of Breslau, as to whether dilute solutions of potassium iodide would undergo decomposition in the presence of free acid. The latter had stated that all hydrous acids would turn paper, moistened with potassium iodide solution, brown. Schönbein, in reply (Jour. für prakt. Chem., 1845, xxxiv., p. 492), pointed out the numerous sources of error from impurities in the iodide, and asserted that had Fischer employed a pure salt, he would have obtained quite different results. Moreover, that atmospheric air, even when mingled with half its volume of carbonic acid, and enclosed in a flask, would not turn a piece of iodide-potassium-starch-paper exposed to its action, blue, however long the exposure might be continued.

It will appear probable, I think, after examination of the experiments detailed below, that Prof. Fischer's results were not due to the cause assigned them, and that both investigators might have legitimately arrived at discordant conclusions from sources of chemical perturbations at that time unsuspected.

A similar remark, perhaps, would apply to the dispute between M. Houzeau and M. L. Sauvage (Compt. Rend., 1868, LXVII., pp. 633, 714, 1138). The former stated that the decomposition noted by M. Sauvage, when he agitated dilute solution of potassium iodide with dilute sulphuric acid, was due to the ether employed to collect the iodine, this ether producing oxygenated water ; and that when chlorform was substituted, no evidences of decomposition could be detected.

Without further anticipating, however, it will be better first to relate the experiments themselves, beginning at the point arrived at in the conclusion of paper in the first number of the current volume, and afterwards deduce certain conclusions, having an important bearing on numerous operations performed in the laboratory.

I.—To determine whether a change of base influenced the rate of decomposition in the sunlight, when the same acid was employed ; or, with the same base, when different acids were used. Ten per cent. solutions of the iodides were employed :

Decompositions effected January 18th. 12—1 P. M.

100 cc	H_2O	+ 5 cc	starch	+ 1 cc	KI	+ 1 cc	H_2SO_4	= 2.25	mgrm.	I.
20 cc	"	"		1 cc	NH_4I	+ 1 cc	H_2SO_4	= 1.35	"	
100 cc	"	"			"		"	= 2.20	"	
20 cc	"	"		1 cc	KI	+ 1 cc	HNO_3	= 0.90	"	
100 cc	"	"			"		"	= 2.00	"	
20 cc	"	"		1 cc	NH_4I	+ 1 cc	HNO_3	= 1.00	"	
100 cc	"	"			"		"	= 2.00	"	
20 cc	"	"		1 cc	NH_4I	+ 1 cc	HCl	= 0.30	"	
100 cc	"	"			"		"	= 1.00	"	

The above experiments were performed with acids whose specific gravities were as follows: Sulphuric acid, 1.84; hydro-chloric acid, 1.202, and nitric acid, 1.4. In the subsequent experiments, the acids employed were some manufactured in the laboratory with especial care, and of such strength, that 1 cc of the sulphuric acid corresponded to 25 cc of a normal soda solution; 1 cc of the hydro-chloric to 10.7 cc, and 1 cc of the nitric acid to 12.6 cc of the normal soda solution.

(Strong sun-light) Feb. 4th, 1879. 1 P. M.—3:30 P. M.

100 cc	H_2O	+ 5 cc	starch	+ 1 cc	KI	+ 1 cc	H_2SO_4	= 16.0	mgrm.	I.
100 cc	"	"		"	1 cc	HNO_3	= 17.5		"	
100 cc	"	"		"	1 cc	HCl	= 16.0		"	
100 cc	"	"	1 cc	NH_4I	+ 1 cc	H_2SO_4	= 14.5		"	
100 cc	"	"	"		1 cc	HNO_3	= 16.5		"	
100 cc	"	"	"		1 cc	HCl	= 13.5		"	
100 cc	"	"	1 cc	CdI_2	+ 1 cc	H_2SO_4	= 11.0		"	
100 cc	"	"	"		1 cc	HNO_3	= 14.5		"	
100 cc	"	"	"		1 cc	HCl	= 11.0		"	

It will be noted that this series shows less of a definite character than the foregoing. The only constant feature being the greater amount of decomposition occurring in presence of the free nitric acid. One phenomenon made strikingly evident in this experiment was, that a great amount of decomposition took place after all the starch present had been precipitated, the supernatant liquid in every comparison-tube being dark yellow. It appeared, therefore, important to keep the starch in excess in each experiment. Repeated :—

Feb. 10th, 1879. Feeble sun-light for 2½ hrs.

100 cc	H_2O	+5 cc	starch	+1 cc	KI	+1 cc	H_2SO_4	= 7.15	mgrm.	I.
100 "	"	5 "	"	1 "	"	1 "	HNO_3	= 5.2	"	
100 "	"	5 "	"	1 "	"	1 "	HCl	= 2.0	"	
100 "	"	5 "	"	1 "	CdI_2	+1 "	H_2SO_4	= 6.5	"	
100 "	"	5 "	"	1 "	"	1 "	HNO_3	= 5.0	"	
100 "	"	5 "	"	1 "	"	1 "	HCl	= 2.1	"	
100 "	"	5 "	"	1 "	KI	+1 "	HNO_3	= 6.4	"	
100 "	"	5 "	"	1 "	"	1 "	HCl	= 2.2	"	
100 "	"	5 "	"	1 "	CdI_2	+1 "	HNO_3	= 5.3	"	
100 "	"	5 "	"	1 "	"	1 "	HCl	= 2.9	"	

In this series, though at the close of the experiment, the supernatant liquids were yellow, yet the amounts of liberated iodine were not so greatly in excess of the required amounts of starch, and more agreement will be found in the results. The experiments were therefore twice repeated, the times of exposure being made so short, that the liquids remained bright blue to the end. Any shade of greenish blue, arising from the admixture of the blue of the starch iodide with the yellow of free iodine, was avoided.

STARCH IN EXCESS.—Feb. 12th, 1879.

I, from 1—1:35 P.M. (sun-light).—II, 3:30—5 P.M. (cloudy).

									I.		II.	
100 cc	H_2O	+5 cc	starch	+1 cc	KI	+1 cc	H_2SO_4	= 0.45	mgrm.	I.	0.25	mgrm. I.
100	"	+5	"	+1	"	+1	HNO_3	= 0.40	"		0.30	"
100	"	+5	"	+1	"	+1	HCl	= 0.15	"		0.10	"
100	"	+5	"	+1	CdI_2	+1	H_2SO_4	= 0.45	"		0.24	"
100	"	+5	"	+1	"	+1	HNO_3	= 0.45	"		0.30	"
100	"	+5	"	+1	"	+1	HCl	= 0.15	"		0.10	"
100	"	+5	"	+1	KI	+1	HNO_3	= 0.55	"		0.30	"
100	"	+5	"	+1	"	+1	HCl	= 0.15	"		0.10	"
100	"	+5	"	+1	CdI_2	+1	HNO_3	= 0.50	"		0.30	"
100	"	+5	"	+1	"	+1	HCl	= 0.15	"		0.10	"

This double series brought out strikingly the fact, which was confirmed in all subsequent trials *with sun-light*, that the amount of iodine liberated in presence of free sulphuric acid, bore a nearly constant ratio, when the same base was combined with iodine, to that occurring in case of hydrochloric acid. The deportment in presence of nitric acid was at variance with that anticipated, on account of a secondary action not investigated until later.

II.—As to the part played by the starch itself in these reactions, the supposition had been, that its affinity for the liberated iodine, though feeble, had nevertheless been one of the agencies at work in determining the fact of a decomposition.

To what extent its influence was exerted, was the object of the next experiment, in which to the contents of four comparison-tubes each containing 100 cc H_2O, 5 cc starch were added (column I), and to four other similarly prepared, no starch (column II.)

Feb. 13th, 1879. 1:15—2:15 P. M. (cloudy).

	I.	II.
1 cc KI + 1 cc H_2SO_4 =	Lost.	1.15 mgrm. I.
1 cc KI + 1 cc HCl =	0.45 mgrm. I.	0.90 "
1 cc CdI_2 + 1 cc H_2SO_4 =	0.55 "	1.15 "
1 cc CdI_2 + 1 cc HCl =	0.45 "	0.90 "

That omission of starch should have increased, and in fact have doubled the amount of iodine set free under these circumstances, was so opposite a result to what was anticipated, that the experiment was repeated in the expectation of finding the former figures incorrect; they were confirmed.

Repeated Feb. 15th, 1879 10:30 A.M.—12:45.

	I.	II.
1 cc KI + 1 cc H_2SO_4 =	3.70 mgrms. I.	7.5 mgrms. I.
1 cc CdI_2 " =	4.00 "	8.2 "
1 cc LiI " =	3.75 "	5.8 "

Further proof was obtained when each of the iodides was exposed with each of the acids, both being employed of same strengths and amounts as stated above, and the solutions made up to 100 cc.

Feb. 19th, time 20 mins. (Without starch.)

1 cc H_2SO_4 + 1 cc KI =	2.85 mgrm. I;	CdI_2 =	3.85 mgrm. I;	LiI =	2.35 mgrm. I.
" HNO_3 " =	2.85 "	" " =	3.15 "	" " =	2.30 "
" HCl " =	2.00 "	" " =	2.45 "	" " =	1.35 "

The decomposition, as will be seen on comparing these results with the foregoing, was so much greater, that after the two following experiments, the use of starch was abandoned in most cases.

III.—Decomposition in Presence of Organic Acids.

To 1 cc of the potassium iodide solution, acetic, tartaric and oxalic acids were added, and the liquids made up to 100 cc. Three of the tubes contained starch—three without. At the end of 2

hours exposure to the sun-light, only the oxalic acid had effected decomposition ; after 8 hours all had changed ; they were left for 2 days, and titrated at the end of 48 hours.

Feb. 22d. ; 2 days. (Organic acids.)

	With starch.	Without.
Oxalic acid	= 6.15 mgrm. I.	8.25 mgrm. I.
Tartaric "	= 0.85 "	0.30 "
Acetic "	= 0.35 "	0.50 "

IV.—Decompositions Effected by the Electric Light.

A cylindrical stand was made, of such dimensions that the centre of the axis of the 100 cc column contained in each comparison-tube, should be at a distance of 6 in. from the focus of the electric light. The electric arc was taken between carbon points, and was produced by electricity generated with a dynamo-electric machine run by steam power, its illuminating capacity varying during the course of the experiment, according to the photometric measurements made by Prof. Morton and Mr. Beckmayer, between 7,000 and 7,500 candles. In the first experiment, the first nine solutions contained 5 cc starch-water, the last two none, and it will be noted that in the latter decompositions were twice as great.

ELECTRIC LIGHT.

	15 min. Mgrm. I.	20 min. Mgrm. I.	7½ min. Mgrm. I.
1 cc H_2SO_4 + 1 cc KI	= 1.65 ; same	= 3.90 ; same	= 3.00
1 HNO_3 + 1 "	= 1.80 ; 1 cc HCl	= 3.15 ; ——	
1 HCl + 1 "	= 1.05 ; same	= 3.40 ; same	= 2.00
1 H_2SO_4 + 1 CdI_2	= 1.80 ; same	= 3.15 ; same	= 2.25
1 HNO_3 + 1 "	= 1.40 ; 1 cc HCl	= 2.30 ; ——	
1 HCl + 1 "	= 1.25 ; same	= 2.50 ; same	= 1.35
1 H_2SO_4 + 1 LiI	= 0.90 ; same	= 3.00 ; same	= 2.10
1 HNO_3 + 1 "	= 0.95 ; ——	——	
1 HCl + 1 "	= 0.60 ; same	= 1.70 ; same	= 1.00
1 H_2SO_4 + 1 KI	= 2.35 ;		
1 " + 1 CdI_2	= 2.30 ;		
1 " + 1 NH_4I	= —— ;	= 2.50 ;	= 2.30
1 HCl + 1 "	= —— ;	= 1.50 ;	= 1.50
1 " + 1 "	= —— ;	= 1.85 ;	= ——

The potassium iodide solution in the first two trials was of ten per cent., in the third twice this amount. The other iodides were of a strength equivalent to the ten per cent. KI solution in each trial.

The highly actinic nature of the electric beam is made strikingly manifest by the large amounts of iodine set free. Moreover, its chemical energy is not the same in the three experiments, twice the amount of decomposition having been effected in the third, as in the second experiment, during the same interval of time. The reason of this variation is not quite clear, for while the light was remarkably uniform during the third experiment, there was no such corresponding difference in the apparent luminous effect.

V.—Comparison of Effects of Sun-light and Electric Light after passing through Absorbing Media.

One cc of a twenty per cent. solution of potassium iodide solution was used in these experiments, with the acids and water. The comparison-tubes were supported in the centers of tall, wide-mouth bottles, so as to be surrounded by an equal thickness (3 cm.) of the absorbing medium on every side. Their tops and those of the bottles were closed in with tin foil, so as entirely to prevent the access of light from that quarter. The blue medium was ammoniacal solution of copper; the yellow, neutral potassium chromate; the red, fuchsine. They were made of such strength that the outlines of an object could be seen through them with equal distinctness, though in each case feebly.

		Sun-light. Mch. 3d, 12 M.—3 P.M.	Electric Light. 20 min.
Blue	1 cc H_2SO_4	= 7.15 mgrm. I.	= 2.00 mgrm. I.
	1 HCl	= 6.65 "	= 1.50 "
Yellow	1 H_2SO_4	= 2.00 "	= 0.165 "
	1 HCl	= 1.35 "	= 0.15 "
Red	1 H_2SO_4	= 4.60 "	= 1.65 "
	1 HCl	= 2.50 "	= 1.00 "

Computed for equal intervals, they would be:

		Sun-light (1 hour).	Electric Light (1 hour).
Blue	1 cc H_2SO_4	= 2.38 mgrm. I.	= 6.00 mgrm. I.
	1 HCl	= 2.22 "	= 4.50 "
Yellow	1 H_2SO_4	= 0.67 "	= 0.50 "
	1 HCl	= 0.45 "	= 0.45 "
Red	1 H_2SO_4	= 1.53 "	= 4.95 "
	1 HCl	= 0.83 "	= 3.00 "

Now, while in the preceding experiments, the action in the electric light had been approximately three times the maximum of that in the sun-light, this ratio, when absorbing media were used, was approached only in case of the blue and red solutions. In other words, the yellow medium absorbed a much larger relative proportion of the actinic rays of the electric, than of the sun-light. Better to understand what occurs in this instance, a study of the absorption-spectra (chemical) of the media, and of the decomposition of the various iodides in the several portions of the spectra of the sun and electric light, has been entered upon.

VI.—Decomposition effected by the Magnesium Light.

The light was furnished by a single-ribbon magnesium lamp, run by clock-work. The reason for repeating the experiment *three* times was that the ratio of decomposition in presence of free hydro-chloric acid was greater instead of less (as when sun and electric light were employed), than in the presence of free sulphuric acid, the acids being of strength before given. This difference is probably connected with the fact, long since recognized in photography, that the maximum of sensitiveness for certain sensitive salts is located in different parts of the spectrum. The potassium iodide was of 20 p. c., the other iodides were equivalent to a 10 p. c. KI solution.

MAGNESIUM LIGHT.

	I.	II. (1 hour). Mgrm. I.	III. ($\frac{1}{2}$ hour.) Mgrm. I.
1 cc H_2SO_4 + 1 cc KI	= lost ;	same = 0.050 ;	same = 0.015.
" HCl "	= 0.225 ;	" = 0.075 ;	" = 0.050.
" H_2SO_4 + 1 cc CdI_2	= 0.080 ;	" = 0.035 ;	" = 0.010.
" HCl "	= 0.150 ;	" = 0.050 ;	" = 0.050.
" H_2SO_4 + 1 cc LiI	= 0.075 ;	" = 0.040 ;	" = indet.
" HCl "	= 0.165 ;	" = 0.050 ;	" = 0.060.
" H_2SO_4 + 1 cc NH_4I	= 0.115 ;	" = 0.060 ;	
" HCl "	= 0.200 ;	" = 0.070 ;	

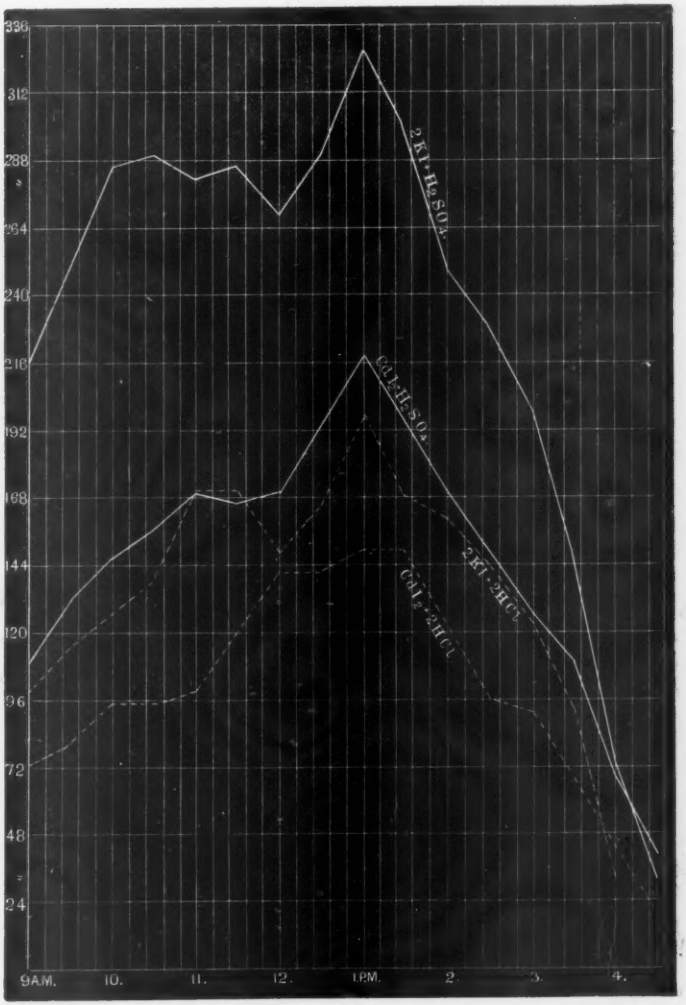
ACTINOMETRIC DETERMINATIONS, FEB'RY 27th, 1879.

	A. M. 9–9:30	9:30–10	10–10:30	10:30–11	11–11:30	M. 11:30–12	12–12:30	P. M. 12:30–1	1–1:30	1:30–2	2–2:30	2:30–3	3–3:30	3:30–4	4–4:30	4:30–5
1 cc H_2SO_4 + 1 cc KI	2.15	2.50	2.85	2.90	2.80	2.85	2.70	2.90	3.25	3.00	2.50	2.25	2.00	1.55	0.75	0.35
1 " HCl "	1.00	1.15	1.25	1.40	1.70	1.70	1.55	1.65	2.00	1.70	1.60	1.45	1.25	0.90	0.35	0.23
1 cc H_2SO_4 + 1 cc CdI_2	1.15	1.30	1.45	1.55	1.70	1.65	1.70	1.95	2.20	1.95	1.70	1.50	1.30	1.10	0.70	0.43
1 " HCl "	0.75	0.85	1.00	1.00	1.05	1.30	1.40	1.40	1.50	1.50	1.30	0.95	0.90	0.65	0.35	0.20
1 cc H_2SO_4 + 1 cc LiI	1.20	1.30	1.35	1.45	1.60	1.50	1.60	1.70	1.55	1.45	1.50	1.40	1.25	1.05	0.65	0.43
1 " HCl "	0.75	0.80	0.95	1.00	1.00	1.00	1.10	1.20	1.20	1.10	1.00	0.75	0.70	0.50	0.30	0.18
1 cc H_2SO_4 + 1 cc NH_4I	1.30	1.40	1.55	1.65	1.80	1.70	1.80	1.90	1.95	1.80	1.70	1.50	1.20	1.05	0.70	0.45
1 " HCl "	0.85	0.95	1.00	1 05	1.15	1.15	1.15	1.20	1.15	1.15	1.10	0.85	0.90	0.60	0 30	0.18

MILLIGRAMMES OF IODINE.

VII. — Actinometric Determination of Sunlight.

In order to compare the decomposition effected by the sunlight, after passing through various thicknesses of the earth's atmosphere, and to compare the relative amounts of iodine set free under these circumstances, when different salts were employed, the determinations summarized in the accompanying table were made. The day was unusually brilliant and unclouded, from beginning to close. The potassium iodide was 20 p. c.; the other iodides were equivalent to one-half this quantity; the acids were of same strength as before given. An inspection of the accompanying diagram, in which the potassium and cadmium curves are given, will exhibit these results more clearly.



MILLIGRAMMES OF IODINE.

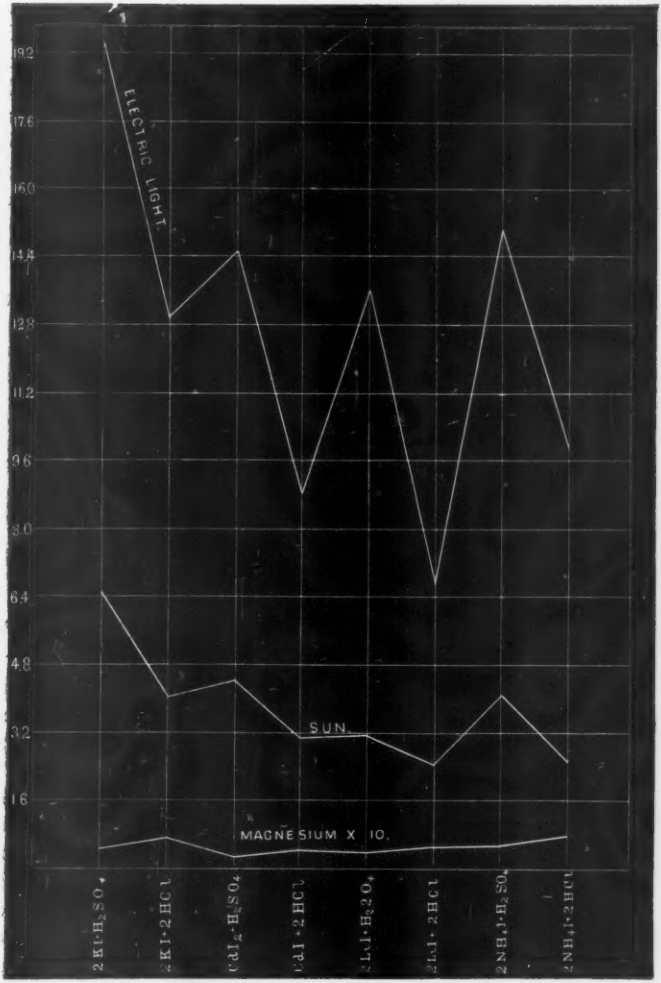
HOURS OF THE DAY.

VIII.—Comparison of Actinic Intensities for Equal Times.

The foregoing results are brought together in the following table, and likewise illustrated in the diagram. The numbers found for the magnesium light have been multiplied by ten, in order that the corresponding curve may be brought into the same illustration with the others.

DECOMPOSITION DURING EQUAL TIMES (1 hour).

	Electric Light.	Sun.	Magnesium.
H_2SO_4 + KI	= 19.5 mgrm. I.	6.5 mgrm. I.	0.05 mgrm. I.
HCl + "	= 13.0 "	4.0 "	0.075 "
H_2SO_4 + CdI_2	= 14.6 "	4.4 "	0.035 "
HCl + "	= 8.8 "	3.0 "	0.050 "
H_2SO_4 + LiI	= 13.7 "	3.1 "	0.04 "
HCl + "	= 6.5 "	2.4 "	0.05 "
H_2SO_4 + NH_4I	= 15.0 "	3.9 "	0.06 "
HCl + "	= 9.8 "	2.3 "	0.07 "


ELECTRIC LIGHT
SUN.
MAGNESIUM X 10.
192
176
160
144
128
112
96
80
64
48
32
16
2KI·H2SO4
2KI·2HCl
CdI2·H2SO4
CdI·2HCl
2LiI·H2SO4
2LiI·2HCl
2NH4I·H2SO4
2NH4I·2HCl

IX.—Influence of Heat upon the Decomposition of the Iodides.

These experiments were made at that period in the investigation, when the effect of light had been recognized, but the concurrent action of oxygen was unknown. The first experiment was made in comparison-tubes, each containing 1 cc KI (of 10 p. c.), 5 cc starch water, and 1 cc H_2SO_4. But I was made up to 20 cc with water, II to 60 cc and III to 100 cc. The times given in the upper line are those at which the change of color first occurred; those in the second line, when the same tint appeared on further heating. The change is quite gradual, running through the scale of colors indicated.

I. (20 cc).

Very Faint Rose.	Faint Rose.	Rose.	Violet.	Blue.
(30°C.) 5 mins.	(33°) 10 mins.	(36°) 16 mins.	(38°) 20 mins.	(45°) ½ hr.
(95°) 95 mins.	(86°) 1½ hr.	(65°) 50 mins.	(56°) 40 mins.	

At boiling point, slowly became colorless.

II. (60 cc).

(32°) 7 mins. (34°) 12 mins. (39°) 24 mins.
(61°) 44 mins. (50°) 39 mins.

Became colorless at 85°, at the expiration of 1 hr. 25 mins.

III. (100 cc).

(34°) 11 mins. (41°) 27 mins.
(50°) 35 mins.

Became colorless at 59°.

In other experiments different figures were obtained, and since the starch is converted into dextrin on heating with dilute acid, its use as an indicator under these circumstances was abandoned.

Effect of Heat in Closed Flasks.—To three flasks, each containing 60 cc H_2O and 1 cc H_2SO_4 were added ½ cc, 1 cc and 2 cc of a 10 p. c. KI solution respectively. They were closed with valve-stoppers, so as to exclude the air, and heated to 93° C. for 6 hours. After cooling, starch was added and they were titrated. The first contained no free iodine, the second gave a very faint rose color to the starch, but too little to titrate, the third contained 0.1 mgrm. I. In other words, when the potassium iodide was diluted 600 times, and heated nearly to the boiling point in a closed flask for 6 hours, the amount of iodide set free was so small that it could not be estimated, and when diluted 1200 times, no change whatever took place.

Effect of Heat in Flasks Entirely Deprived of Air.—Three comparison-tubes were connected in the manner of wash-bottles. Each

contained 100 cc water and 1 cc KI solution of 20 p. c., but the first had 1 cc H_2SO_4, the third 1 cc HCl as well. After passing a stream of carbonic anhydride, previously washed with KI solution, through them for ½ hour, they were immersed in a salt bath and the temperature raised to 105° C. After two hours exposure, a current of carbonic anhydride constantly flowing, no iodine had been liberated in either of the three vessels. Similar acidified solutions, heated to the same temperature, in contact with air, turned yellow immediately.

X.—Reactions Involved in the Decomposition of Iodides.

The fact of the decomposition of the dilute iodides in presence of free acid having been established, it was essential to explain the accompanying reactions. The following might be legitimately conceived of :

[1.] $2\,KI + 2\,H_2SO_4 = K_2SO_4 + 2\,H_2O + SO_2 + I_2$.
[2.] $4\,KI + 2\,H_2SO_4 + O_2 = 2\,K_2SO_4 + 2\,H_2O + 2\,I_2$.
[3.] $4\,KI + 4\,HNO_3 + O_2 = 4\,KNO_3 + 2\,H_2O + 2\,I_2$.
[4.] $2\,HNO_3$ (in sun-light) $= N_2O_3 + H_2O + O_2$.
[5.] $2\,KI + 2\,HNO_3 + N_2O_3 = 2\,KNO_3 + H_2O + N_2O_2 + I_2$.
[6.] $4\,KI + 4\,HCl + O_2 = 4\,KCl + 2\,H_2O + 2\,I_2$.

It was evident the equation [1], which is true of concentrated solutions, could not apply to these very dilute ones, for two reasons : 1st. Because, as a fact, no sulphurous acid was given off. 2d. Because free sulphurous acid and free iodine could not co-exist in the same solution. It was, therefore, necessary to adopt the supposition contained in the second equation, the required oxygen being supplied by the air. No such alternative was possible in the case of hydrochloric acid. And with nitric acid, since it had been found (see preceding paper) that nitric acid contained in hermetically sealed flasks and exposed to sunlight, contained much nitrous acid, besides a primary decomposition, similar to the foregoing, a secondary reaction was not only possible, but probable. To this secondary reaction, the amount of which could be readily foretold, the high results obtained with nitric acid were attributed.

Effect of Oxygen in Sun-light.—To test the above hypothesis, two opposed methods were desirable—a negative, when the iodides in the entire absence of oxygen, should undergo no change, and a positive, when the iodides in presence of excess of oxygen, should change at a correspondingly accelerated rate. No method of carrying into execution the first method was found very feasible, except that of replacing the air held in solution and the atmosphere itself,

by another gas. In this case, carbonic acid, which was employed for the purpose, appeared especially suitable, since it was in the presence of free acid, that the experiments were to be performed. Three comparison-tubes were filled up in the manner of wash-bottles, the joints being perfectly air-tight. 100 cc water and 100 cc potassium was put in each. The first contained as well, 1 cc sulphuric acid, the third, 1 cc hydrochloric acid, the middle tube no acid. After displacing air in solution by a current of washed carbonic acid continued for three hours, the tubes were exposed at a sun-window, and kept there during 48 hours, the sun shining upon them during the whole of two days. At the end of this time, a slow current of the gas having been kept flowing through the solutions, they were found to have experienced no change. Without changing the contents of the tubes, or any of the arrangements further than replacing the stream of carbonic acid by a current of oxygen, the exposure was continued, when a change quickly began, and at the expiration of 2¼ hours, during much of which time the sun was overclouded, the two solutions containing free acid had become deep yellow, the one without was entirely colorless. On titration, the first was found to contain 12.1 mgrms. of free iodine, the third 9.5 mgrms., the middle tube none.

These experiments put in a striking light the *role* which oxygen plays in the decomposition of iodides in presence of certain free acids. But two other points which they likewise render conspicuous should not be overlooked—that solution of pure potassium iodide, even when exposed to sun-light, is permanent not only in presence of excess of oxygen, but likewise in presence of excess of carbonic acid. These two points are important in their bearing upon ozonometry.

Effect of Air or Oxygen in the Dark.—When air had been completely replaced by carbonic acid, no decomposition occurred. Otherwise, not only did the rate of decomposition increase with the concentration (as shown in a preceding paper), but did not fall off to zero, even when the acid had been diluted four thousand, and the iodide forty thousand times. At the expiration of five days, with solutions one-half so dilute as those above (1 cc acid and 1 cc KI solution, in two liters), the iodine set free amounted to 0.25 mgrm.

Conclusion.—It is evident that the above results necessitate the use of corresponding precautions and modifications, in cases where potassium iodide is employed in titration, more especially in the determination of nitrates in potable waters, acid solutions, etc. ; but the work instituted in this direction has not as yet been completed.

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(*See Proceedings March, 1879, p. 37.*)

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